User Manual

Helijet

Helium jet for X-ray crystallography

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Version 2.0

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Important Information

This manual is intended to help the user operate the Helijet system manufactured in Poland by Oxford Diffraction.

Product:	Helijet
Serial number:	HAA-003/01 onwards
Electrical Ratings:	1/N AC, 100-240 V, 50/60 Hz, 120 W

Before attempting to operate the system, PLEASE READ THE INSTRUCTIONS.

Ensure that this manual is kept with the system for its entire lifetime. If the system is sold or given to someone else, pass the manual on to the new user of the system so that they are aware of the potential hazards associated with this equipment.

This product should only be used by persons legally permitted to use it.

If the equipment is used in a manner not specified in the User Manual, the protection provided by the equipment may be impaired. The warranty may be affected if the system is misused, or the recommendations in this manual are not followed.

Important Health and Safety Notice

When returning a component for service or repair, the component must be shipped with a signed declaration that the product has not been exposed to any hazardous contamination, or that appropriate decontamination procedures have been carried out so that the product is safe to handle.

Care has been taken to ensure the information in this manual is accurate and at an appropriate level. Please inform Oxford Diffraction if you have any suggestions for corrections of improvements to this manual.

Service and support is available for technical and operational issues as indicated below.

- **E-mail**: support@oxford-diffraction.com
- **Phone**: +44 (0) 1235 532132 between 8 a.m. and 4.30 p.m. (UK time), Monday to Friday

This user manual has been written according to standard 89/392/EEC and further modifications.

Helijet and Xcalibur are trademarks of Oxford Diffraction Limited in some jurisdictions and registered trademarks of Oxford Diffraction Limited in other jurisdictions.

Oxford Diffraction acknowledges all trademarks and registrations.

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1. Health and Safety Information

1.1. General

In normal operation the system is designed to operate safely. All users of the equipment should be aware of potential hazards which exist in and around equipment of this type and the ways of avoiding possible injury and equipment damage which may result from inappropriate ways of working. A description of potential hazards, and how to avoid them, is given in this section.

If you are in doubt about any aspect of the operation of the system contact a local expert or your supplier.

This manual adopts the following convention:

WARNING
Indicates a potential hazard which may result in injury or death

CAUTION
Indicates a potential hazard which may result in damage to equipment

Warning symbols on the equipment are:

Protective conductor terminal

Earth (ground) terminal

CAUTION
Risk of electric shock

CAUTION
Refer to accompanying documents

See original manufacturers' manuals for further safety data on third party equipment supplied with the system. A list of these is given in this manual.

WARNINGS
Do not take risks. You have a responsibility to ensure the safe condition and safe operation of equipment.
Do not work alone. Working alone is dangerous as there is no one to help if you have an accident.

1.2. Electrical Safety

In normal use users are protected from the dangers associated with the voltage, current and power levels used by this equipment. Only personnel who are qualified to work with the voltage, current and power levels used by this equipment should attempt to perform service work on this equipment.

1.2.1. Potential Electrical Hazards

The following list is not intended as a complete guide to all the electrical hazards on the system, but serves to illustrate the range of potential hazards that exist:

- electric shock
- electric burn
- fire of electrical origin
- electric arcing

1.2.2. Recommended Precautions

⚠ **WARNING**
All of the electrical equipment supplied as part of the system should be provided with a protective ground. Do not remove protective grounds as this may give rise to an electrical safety hazard. It is vitally important that the system is properly grounded at all times.

Follow local and national electrical regulations and procedures.

Do not defeat interlocks, remove connectors, disconnect equipment, open safety covers, dismantle or modify equipment unless you are qualified and authorised to do so and you are fully conversant with its operation and potential hazards or have total assurance through your local electrical permit to work system that the equipment has been made safe.

Make sure that the mains supply is fused at an appropriate rating and that it can be isolated locally via a clearly labelled, clearly visible and easily accessible isolating switch. Isolate the supply before carrying out any maintenance work.

1.2.3. First Aid

⚠ **WARNING**
Do not attempt to administer first aid to someone who may have suffered electric shock until the source of the shock has been isolated.
Mains voltages are present in the system. These can cause serious injury or death.
Only personnel qualified and experienced to work with such currents and voltages should perform service or maintenance work on this equipment.

A course in first aid to include methods of artificial respiration is recommended for those whose work involves equipment which may produce a high voltage.

1.3. Mechanical Handling Safety

WARNING
Lifting points are provided for safe handling of components and safe handling practice must be observed to comply with local regulations. Check that lifting points are used only for the job intended.
The system itself and some components are heavy and require careful handling. Use safe lifting procedures for heavy items to prevent possible strain injury.

1.4. Safe Mechanical Practice

In normal use personnel are not required to undertake mechanical work. However, servicing or repair may necessitate access to any part of the system. Only suitably qualified personnel should attempt to dismantle, modify or repair equipment.

1.5. Vacuum

WARNING
Protect the vacuum spaces in cryogenic systems with an overpressure relief valve for the following reasons:

- **A small air leak may go unnoticed. Air leaking into the vacuum space may freeze onto cold surfaces or be absorbed by a sorption pump. When the system is warmed up after an extended period the frozen air may expand to fill the vacuum space to a pressure greater than it can safely withstand.**
- **If a vessel filled with cryogenic fluid is damaged, the fluid may be released into the vacuum space, so the vacuum fails and the fluid warms up rapidly. Large relief valves are required on the liquid and vacuum spaces to vent the gas generated.**

CAUTIONS
Do not evacuate vessels that are not designed to work under vacuum. There is a danger that the vessel or tube may implode.

Only vent vacuum vessels slowly to avoid damaging the system. The shock of the sudden pressure increase may cause an otherwise safe tube to collapse. Some systems must be vented slowly to allow the pressure to equalise in different parts of the system.

1.6. X-ray Radiation

WARNING
This equipment is used on systems that incorporate an X-ray source. Ensure that safe working practices relating to radiation are employed. Follow any local, national or international rules and guidelines.

For use in the UK adhere to the Ionising Radiations' Regulations 1999. For countries outside the UK the appropriate laws apply such as registration and inspection. Be aware of your duty of safety to your employees and visitors.

1.7. Cryogenic Temperatures and Fluids

All personnel operating this equipment must have received proper training from a competent person in working with cryogens as all cryogens are potentially hazardous. Liquid helium (LHe) is less dangerous than some other cryogens because it is neither poisonous nor flammable. Do not ignore the recommended precautions as accidents can cause blindness or death and even small burns are extremely painful and take a long time to heal.

The hazards associated with handling cryogens include:

- Extreme cold and the risk of cold burns or frostbite
- Asphyxiation (due to displacement of atmospheric oxygen)
- Fire and explosion hazards (through oxygen enrichment)

Signs that a hazard might be developing include:

- Unusually high (or low) boil off
- Unusual condensation of atmospheric moisture
- Unexpected patches of frost on the outside of the cryostat, transfer tube or storage dewar
- Faulty valves.

If you suspect that there is a fault with your system, warm it to room temperature and repair the fault immediately, without waiting to finish your experiment. The fault may lead to additional unknown hazards.

Further information about cryogenic safety can be found in the *Cryogenics Safety Manual - a guide to good practice* by the British Cryogenics Council, (ISBN 0-85298 5010).

1.7.1. Protection from Extreme Cold

WARNINGS

- **Label vessels clearly to indicate their contents so that others can also take appropriate precautions.**
- **Wear protective clothing.**
- **Do not wear wet clothing - it could freeze to your skin.**
- **Wear goggles to protect your eyes.**
- **Use loose fitting gloves so that you can remove them easily if you spill liquid inside them**
- **Wear overalls or similar clothes, preferably without pockets or turn-ups**
- **Wear sensible shoes (not sandals) and ensure that trousers cover the top of your shoes to prevent spilt cryogens running into your shoes.**
- **Only use suitable metal tubing to transfer cryogens. Do not use rubber, silicone rubber, or plastic tubing. The use of polythene and nylon is not recommended although they are sometimes used. Only used materials approved for cryogenic use by the manufacturer. Carefully test any materials to be used in safe conditions first.**
- **Only use containers specifically designed for use with particular cryogens as many materials (even some common steels) become dangerously brittle at low temperatures.**
- **Tie bungs (or stoppers) to the top of the containers so they do not get lost, or get blown out by high pressure and become dangerous projectiles.**
- **Handle cryogens carefully. Cryogens boil violently and splash when they come into contact with warmer objects.**

Very cold objects can stick to bare skin (by rapid freezing) and tear away the skin. This freezing process can occur rapidly.

When inserting an open-ended pipe into a cryogenic liquid, block off the warm end until the other end has cooled down, otherwise cold liquid is likely to squirt out of the open end under self-generated pressure. Never point pipes towards someone else.

CAUTIONS
Protect equipment from extreme cold. When objects become very cold they may become brittle and fracture easily.

The following examples illustrate the damage that can be caused by extreme cold:

1. Cryogens spilt on vacuum equipment may freeze vacuum seals causing loss of the insulating vacuum. Do not allow the top flange of a cryostat to get too cold (warm it with a hot air blower if ice starts to collect).
2. Cryogens spilt on electrical cables may make the insulation freeze and fracture thus causing an electrical hazard. Do not keep cables on the floor where cryogens may be spilt on them.
3. Spilt cryogens can also condense moisture from the air to form a thick mist which can obscure your vision. If you are enveloped in a cloud of cold gas you may lose your balance and fall. This is particularly dangerous if you are standing on a ladder.
4. Spilt cryogens can damage floors. In particular, plastic tiles may become very brittle, or crack.

1.7.2. Protection from Asphyxiation

WARNING
Only store or use cryogens in a well ventilated room. If all or part of the oxygen is removed from the atmosphere you may become unconscious without warning. To protect yourself against asphyxiation:

- **Ensure that there is sufficient ventilation in your own laboratory and in other rooms nearby**
- **Install sensors which will sound an alarm if the oxygen level is too low, unless you are sure that the room is well ventilated**
- **Leave the room immediately if a large amount of cold gas is released quickly**
- **Leave the room immediately if a large amount of liquid is spilt. Consider sounding the fire alarm if there is likely to be a fire hazard, or to clear the area quickly**
- **If there is a possibility of a lack of oxygen in the room, hold your breath, to remind yourself of the urgency of leaving the area**
- **Do not accompany storage or transport vessels in confined spaces (especially in lifts, elevators or enclosed vehicles)**
- **Use a suitable exhaust system to pipe exhaust gases away from the cryostat to the atmosphere or into a helium recovery system**
- **If you store cryogenic liquid vessels in a room that is not well ventilated, put warning signs on the doors so that no one enters the room until it is well ventilated. Lock the room and check the oxygen concentration before anyone enters the room**

Remember that cold helium gas tends to collect near the ceiling.

The effects of asphyxiation depend on the oxygen concentration (the acceptable oxygen concentration level is normally 18-22%). There is no sensation of breathlessness to warn you that you are being asphyxiated; breathlessness is a symptom of a high concentration of CO_2 (not a low concentration of oxygen). If the oxygen level is being reduced slowly the first symptoms may be increased pulse and breathing rate, with impaired judgement, but the very first symptom you notice may be that you cannot stand up or even crawl. By this stage it is already too late for you to help yourself.

1.7.3. Fire Hazards

Most of the fire hazards encountered in laboratory scale cryogenic systems are caused by oxygen enrichment which can cause spontaneous combustion.

WARNINGS

To avoid the risk of fires due to spontaneous combustion caused by oxygen enrichment:

- **Ensure that there is no oil or grease in a position where it may be exposed to liquid air (even if it is liquefied by accident)**
- **Do not smoke and forbid smoking in the areas where cryogens are handled**

If a fire does occur ensure that can be extinguished promptly by

- **Ensuring that suitable fire extinguishers are available**
- **Training people to use the fire extinguishers properly (using a fire extinguisher incorrectly could block exhaust ducts from the cryostat so that the pressure inside becomes dangerously high).**

Liquid oxygen can condense from the air onto surfaces which are at temperatures below 90 K. You can often see liquid air running from a cold helium recovery line if a helium transfer is carried out too quickly.

If a fire breaks out, sound the fire alarm, and ensure that everyone leaves the area. Special expertise is required to put out these fires safely, so if you have not been trained how to do it, find someone who has. Using the wrong type of fire extinguisher, or not using it properly, may block the exhaust vents of the cryostat with ice; if the exhaust gases cannot escape through a suitable relief valve the system will probably explode.

After the fire has been extinguished ensure that the system is safe.

1.7.4. Protection Against Explosion and Blockages

These guidelines only apply to preventing explosions caused by accidentally blocking the exhaust of a cryostat, or warming up a cryostat which has accidentally condensed contamination from the atmosphere onto cold surfaces.

WARNING
If the exhaust ports are connected to pumping lines or a helium recovery system, Ensure that the lines are large enough for the expected gas flow. The diameter of the tube should be at least as large as the diameter of the exhaust port.

The helium reservoir exhaust must be fitted with a non-return valve or connected to a helium recovery system to prevent ambient air leaking back into the cryostat. The valve should be at least large enough to handle the normal gas flow during a liquid helium transfer.

WARNINGS

1. **Fit large pressure relief valves to systems to allow the helium gas to leave the system quickly. If your system was supplied with one of these valves, never cool the system down without it. If a major vacuum failure occurs the evaporated helium will vent safely through the valve(s).**

2. **All relief valves on the system must be large enough to handle the maximum possible gas flow, caused by all the different failure modes happening together. Failure of the insulating vacuum can make all the liquid in the cryostat evaporate very quickly.**

3. **Keep all of the system's exhaust vents clear of ice.**

WARNING

1. **Check for blockages regularly and often.**

2. **Check the system boil off regularly. If there is no boil off and you know that the system is not empty check whether a blockage is preventing the natural boil off. The pressure inside the cryostat will rise until it reaches a dangerously high level.**

Even after all the liquid has evaporated there should still be a perceptible flow of exhaust gas at the gas in the system warms up. Even if the system contains no liquid, it is not ‘empty’ until it reaches ambient temperature.

Consider the explosion hazard when you are warming up the system, especially if it has been cold for a long time. Small leaks may go unnoticed, and any air that leaks into the vacuum spaces may then be cryo-pumped onto the cold surfaces. This expands to form a large volume of gas as the system warms up.

1.7.5. Liquid Helium - Specific Techniques

Liquid helium may cause blocked vents or oxygen enrichment. As liquid helium is the coldest of all cryogenic liquids it will condense and solidify any other gas coming into contact with it. Any surfaces cold enough to condense air in normal operation could also increase the oxygen concentration to a dangerous level, and they should be cleaned to 'oxygen clean' standards. This is why liquid helium containers are often labelled "Flammable liquid" even though the liquid is not flammable.

Insert warm objects into liquid helium vessels very slowly to ensure that they are well cooled by the cold gas before they reach the liquid and:

- Reduces the hazard from rapid boiling which produces a jet of cold gas
- Considerably reduces the consumption of liquid, saving money

Always make sure that one end of a warm tube is closed before you lower it into liquid helium.

WARNING
Liquid helium must be kept in specially designed storage or transport vessels.

Ensure that dewars have a non-return valve fitted in the helium exhaust line at all times, or are connected to a helium recovery system, so that air does not enter the neck and block it with ice.

If possible, keep liquid helium at a slight positive pressure, so that if there is a leak, helium may leak out but air does not leak in.

If you transfer liquid helium into a liquid nitrogen shielded dewar check that the nitrogen vents are clear. The cold helium gas sometimes cools the liquid nitrogen reservoir to below 77 K, (sometimes called 'supercooling'), so that it starts to condense gas from the air. The water vapour in the air then freezes in the vents, and may block them. Always make sure that at least one non-return valve is fitted to these vents.

WARNING
To avoid safety hazards, make the following daily checks on liquid helium vessels:

- **Check the boil off and investigate if it is higher or lower than expected**
- **Ensure that the non-return valve (or recovery system) is still fitted to the exhaust**

1.7.6. First Aid

If any cryogenic liquid comes into contact with eyes or skin, immediately flood the affected area with large quantities of cold or tepid water. Never use hot water or dry heat, which could cause further burning to the damaged area. If the skin is blistered, the burns are extensive, or the eyes have been affected, seek medical advice immediately.

Cover burns with sterile dressings. Do not allow the victim to smoke or drink alcohol. Remember that there is a risk of creating a second injury if you put a frozen finger into your mouth to warm it up. The delicate tissues inside your mouth could be frozen too.

If the victim's lungs have been exposed to cold gas enough to cause distress, or if in doubt, take him or her to hospital immediately. If the victim is suffering from dizziness or loss of consciousness due to asphyxiation:

- Ensure that you are safe first (and in some cases this means that you should not enter the area without breathing apparatus)
- Summon medical help immediately
- Move the victim to a well-ventilated area if it is safe to do so

Apply artificial ventilation or resuscitation if necessary.

1.8. High Pressures

WARNING
Know the law about high pressure gas cylinders and follow it. High pressure cylinders are often used to store gases (typically at pressures up to 200 bar). Most countries have laws about using them.

- **Chain cylinders to a fixed object or keep them in specially designed trolleys**
- **Only use approved and tested high pressure fittings**

Gas cylinders become dangerous projectiles if they are ruptured or the valve is knocked off. They can break through thick walls or travel hundreds of metres (by rocket propulsion).

1.9. Modifications and Service

The safety, reliability or performance of the equipment may be impaired if assembly operations, extensions, re-adjustments, modifications or repairs are not carried out in accordance with the instructions provided in this manual and with any other instructions issued by the manufacturer. If you wish to modify the equipment please contact Oxford Diffraction for further advice.

It should be stressed that those parts of the equipment which are interchangeable, and which are subject to deterioration during operation, may significantly affect the safety of the equipment.

2. Introduction

2.1. Scope

This manual applies to the Helijet designed and manufactured by Oxford Diffraction.

2.2. How to Use This Manual

This manual is intended to provide operators with a practical guide to the system and its operation. This is intended to familiarise the user with how the system works and provide a better understanding of the system operation.

All personnel who are likely to operate the system or come into contact with any of the system components should read the **SAFETY** section of the manual. This provides basic information aimed at highlighting the safety hazards associated with the equipment.

More detailed information and instructions for component parts of the system are given in the third party manuals supplied with the system, which are listed in this manual. These manuals should also be read and understood before operating the system.

The purpose of this manual is to:

- explain how to operate the equipment
- explain how to interface to the equipment
- list performance characteristics of the equipment
- describe how the equipment operates
- assist with simple fault finding and maintenance

2.3. System Description

Helijet is an open flow helium attachment for an X-ray diffractometer. It provides a flow of cold (less than 15K) helium gas to the crystal sample without requiring any shielding around the crystal.

The Helijet is designed for cryo-crystallography experiments. It has been developed for use with any desk-top X-ray diffractometer, particularly the Xcalibur diffractometer, and also for use on the end stations of synchrotron beamline.

The Helijet is intended for indoor use in a laboratory or research environment.

3. Specifications

3.1. Environment

Ambient temperature	0°C to 35°C
Storage temperature	0°C to 40°C
Relative humidity	10 – 80% non – condensing
Ventilation	Well ventilated room to allow helium gas to escape freely into the atmosphere
Flooring	Smooth, even flooring to allow helium dewar to be positioned easily

3.2. Space Requirements and Clearances

Maximum distance from sample to centre of helium storage dewar during operation	250 cm
Helium storage dewar diameter (size of dewar used depends on length of experiment)	About 70 cm diameter for a 50 l dewar About 2 m diameter for a 1000 l dewar
Minimum clearance above top of dewar	1.5 m

3.3. Performance Data

Operating temperature range	<15 K to 100 K
Distance from tip of nozzle to sample	5 mm
Crystal shielding	None required

3.4. ITC 502 Data

Sensor type	Chromel/Au – 0.07% Fe T/c
Sensor data range (on temperature controller)	tG.7b.ccf

3.5. Services Required

Liquid Helium

Liquid helium consumption	2 l/h
Liquid helium required for cooldown and at end of experiment	6 l

Helium Gas

Purity	99.99%
Minimum pressure	0.5 barg
Flow rate	30 l/min @ ATP

Electricity

Number of outlets	2
Number of phases	1
Voltage	100 to 240V.
Maximum current	1.7A per controller
Earthing	No special earthing is required.

3.6. Third Party Equipment

Auto Needle valve	Oxford Instruments
Pump	Pfeiffer MVP0202-3 AC
Temperature controller	Oxford Instruments ITC 502
Liquid helium transfer tube	Oxford Instruments LLT series

4. Technical Description

4.1. Overview

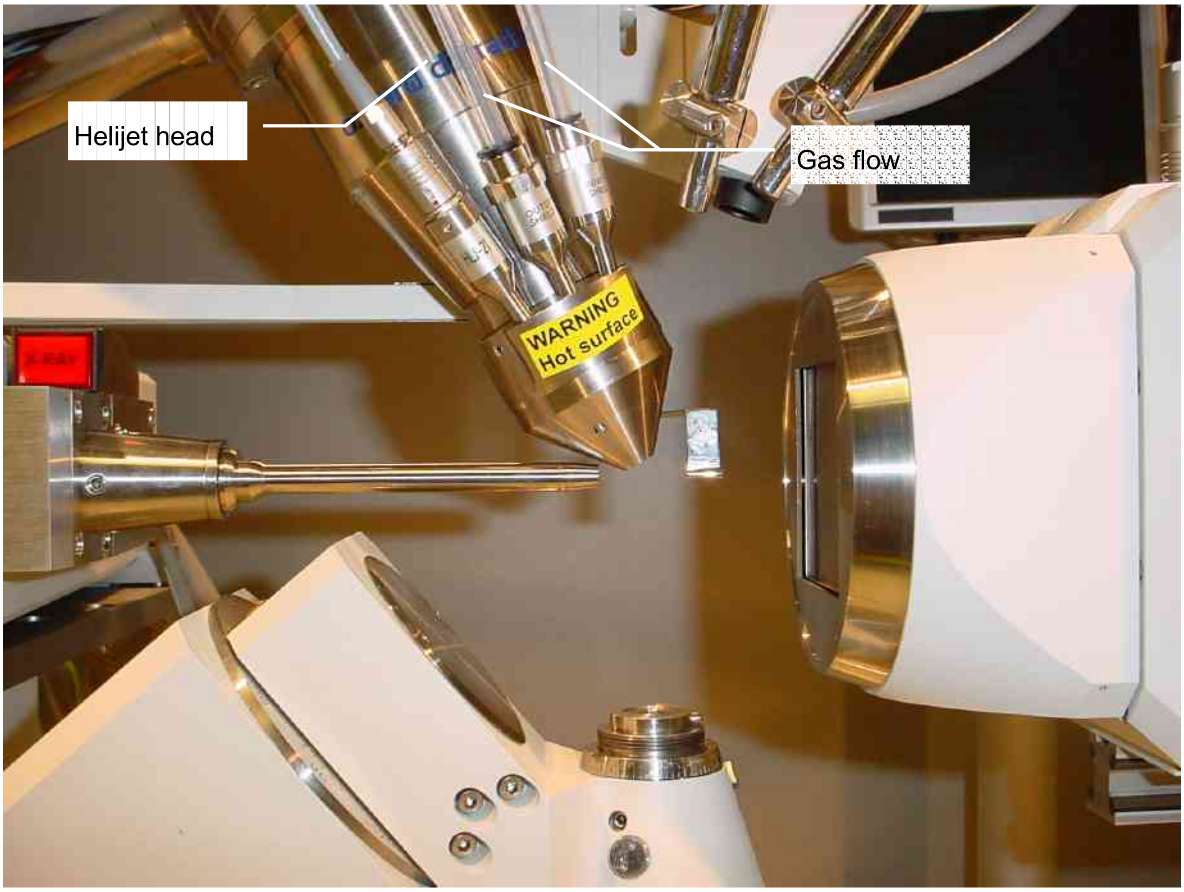


Figure 4-1 View of the Helijet Head Installed on an Xcalibur Diffractometer

Helijet is an open flow helium attachment for an X-ray diffractometer. It provides a flow of cold (less than 15K) helium gas to the crystal sample. It has a very uniform temperature distribution across and along the main stream. No shielding is required around the crystal, so the kappa goniometer can use all its degrees of freedom for to orient the crystal. There is also clear visual access to the sample (crystal).

The Helijet system consists of the following items:

- Helijet head
- Cryogen transfer tube
- Gas flow controller (with integral pump, N/V, and gauges)
- Counterflow helium transfer tube
- Helium dewar (this item may not be supplied with all systems, but is required for the operation)

The diagram below shows a schematic diagram of the Helijet system installation.

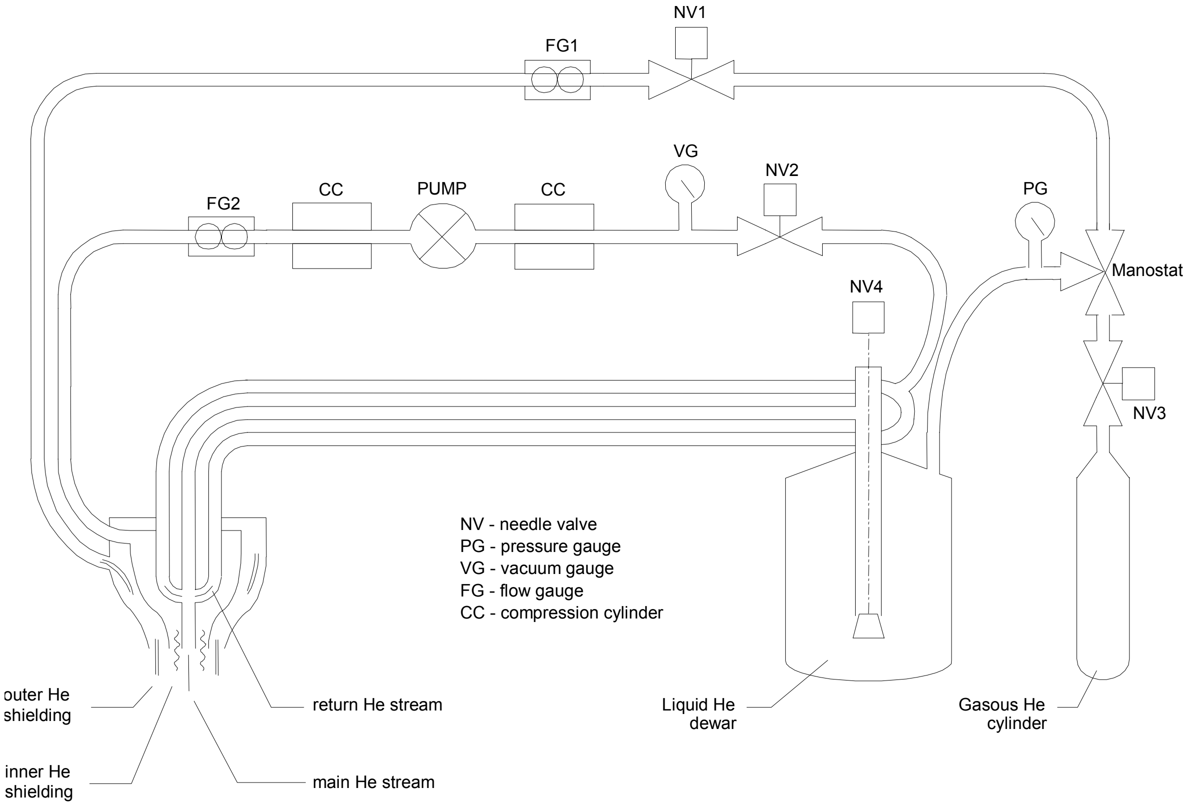


Figure 4-2 Schematic Diagram of Helijet System Installation

4.2. Principles of Operation

The main gas stream (from the dewar to the exit nozzle in the Helijet head), is thermally isolated from the surroundings by the enthalpy of the returning helium gas stream. The first stream surrounding the main stream of cooling gas flows in the opposite direction to the main stream. This permits the Helijet to operate in two different modes simultaneously: as a continuous flow cryostat and as an open system.

4.2.1. Operation as an Open System

In this mode part of the main stream cools the sample. The other part of the main stream reverses direction inside the Helijet head and flows back to thermally isolate the vacuum system surrounding the main stream. Partially warmed gaseous helium is directed to the HELIJET head to create a coaxial shielding stream around the end blowing nozzle of the HELIJET.

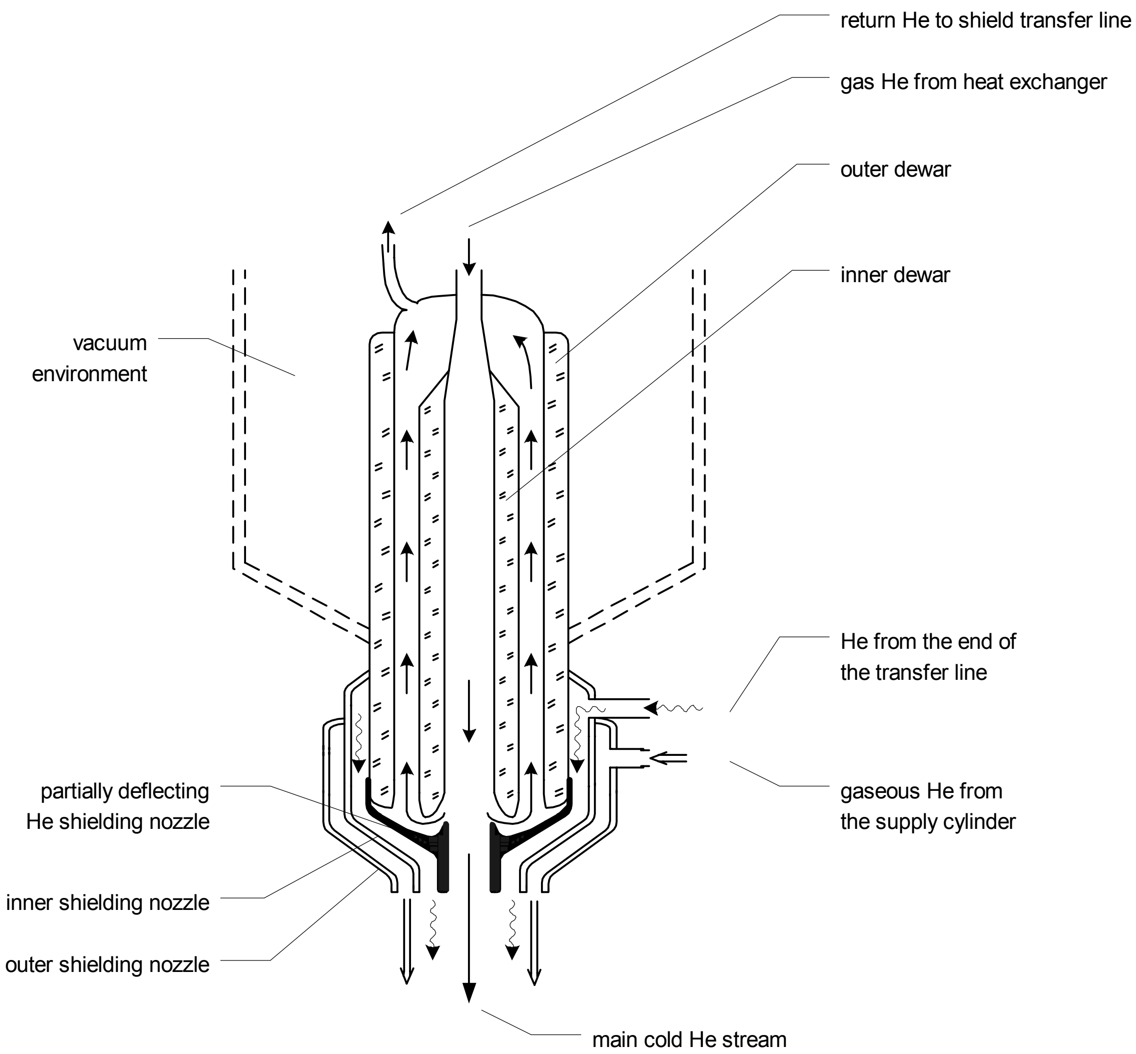


Figure 4-3 Schematic Diagram of the Helijet Head

Additionally, high pressure helium gas forms a coaxial curtain around both the inner streams. This reduces the environmental influence on the stability of both helium streams (the main stream and the shielding one).

The pump, in conjunction with needle valves NV1 and NV2, has two functions. It creates a low pressure to facilitate the reverse flow of the protective stream, and simultaneously pressurises the main dewar containing liquid helium, thus pushing the helium towards the Helijet head. The dual-way valve has two functions: to open the system to the helium gas during start-up and to pressurise the dewar during operation.

The permanent flow of the shielding stream can be changed and measured by the flow gauge FG1.

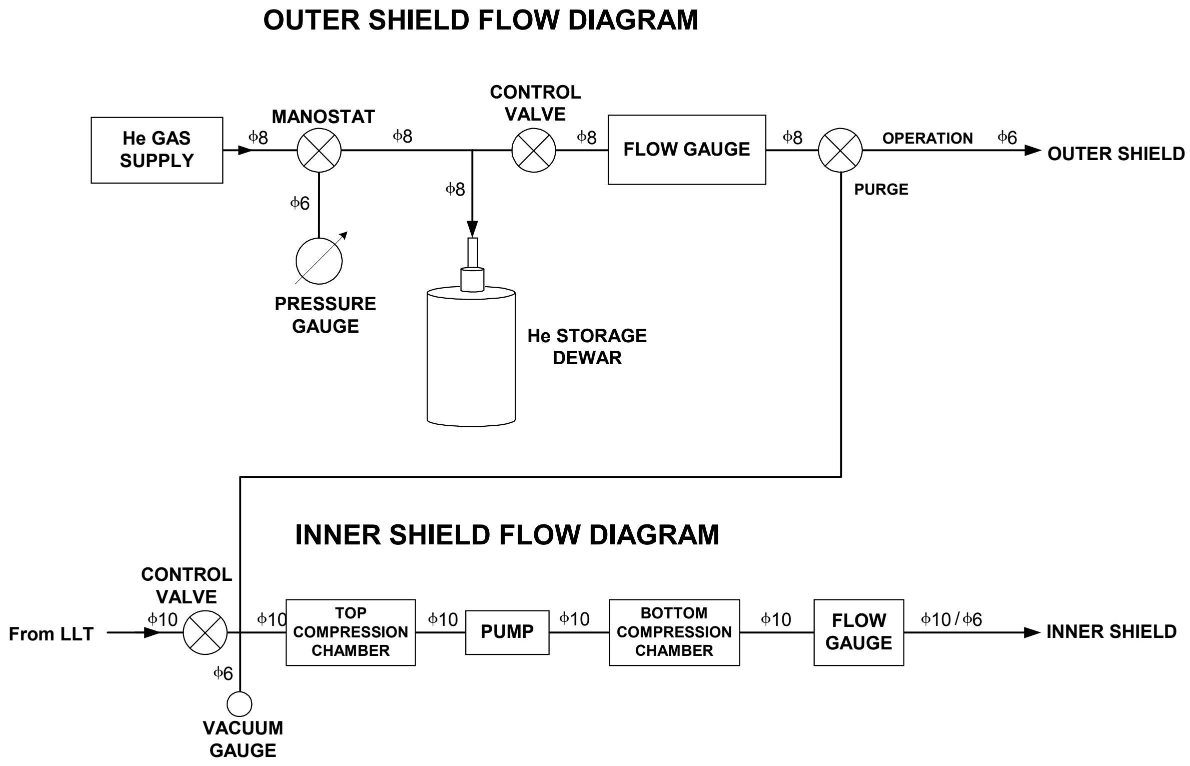


Figure 4-4 Flow Diagram

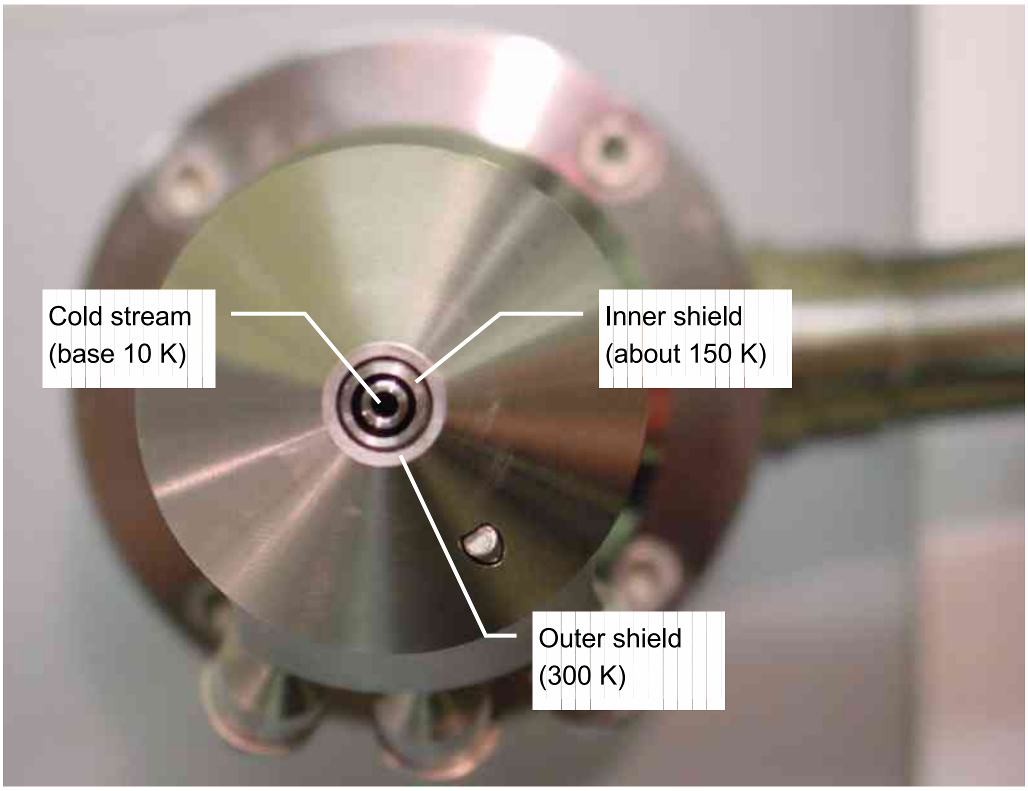


Figure 4-5 View of the Helijet Head Looking Towards the Nozzle

4.3. The Helijet Head

The Helijet head does not have an internal reservoir to store a supply of cryogens. The liquid is supplied from a separate storage vessel through an insulated transfer tube. It flows through a heat exchanger, through to the sample space. A proportion of the gas is diverted out of the cryostat to the pump located in the GFC1. A heater is mounted on the heat exchanger, and this can be used with a temperature controller to balance the cooling power of the cryogen and to control the temperature of the gas before it reaches the sample space.

It is recommended that only liquid helium be used in this cryostat. Temperatures down to about 10 K can be reached using liquid helium. It is possible to maintain a temperature of about 10 K continuously using the standard gas flow controller (GFC1).

4.4. The Gas Flow Controller (GFC)

The Oxford Diffraction GFC1 gas flow control unit is used to promote the flow through the Helijet head and transfer tube. It contains an oil free diaphragm pump with a nominal displacement of 1.2 m^3/hr. The air leak rate is guaranteed to be less than 1x 10^{-1}mbar l/s. This pump is described fully in a separate manual.

The GFC includes flow meters (calibrated for helium liquid equivalent), a pressure gauge and a vacuum gauge. These instruments allow the performance and operation of the complete system to be monitored.

There are 2 gas flow circuits within the system, the inner shield flow and the outer shield flow.

The inner shield flow is created by the internal diaphragm pump and can be controlled by the manual control valve situated on the left-hand side on the front panel of the GFC1 unit.

The outer shield flow is supplied by the room temperature helium gas and is controlled by the manual control valve located on the right hand side on the front panel of the GFC1 unit.

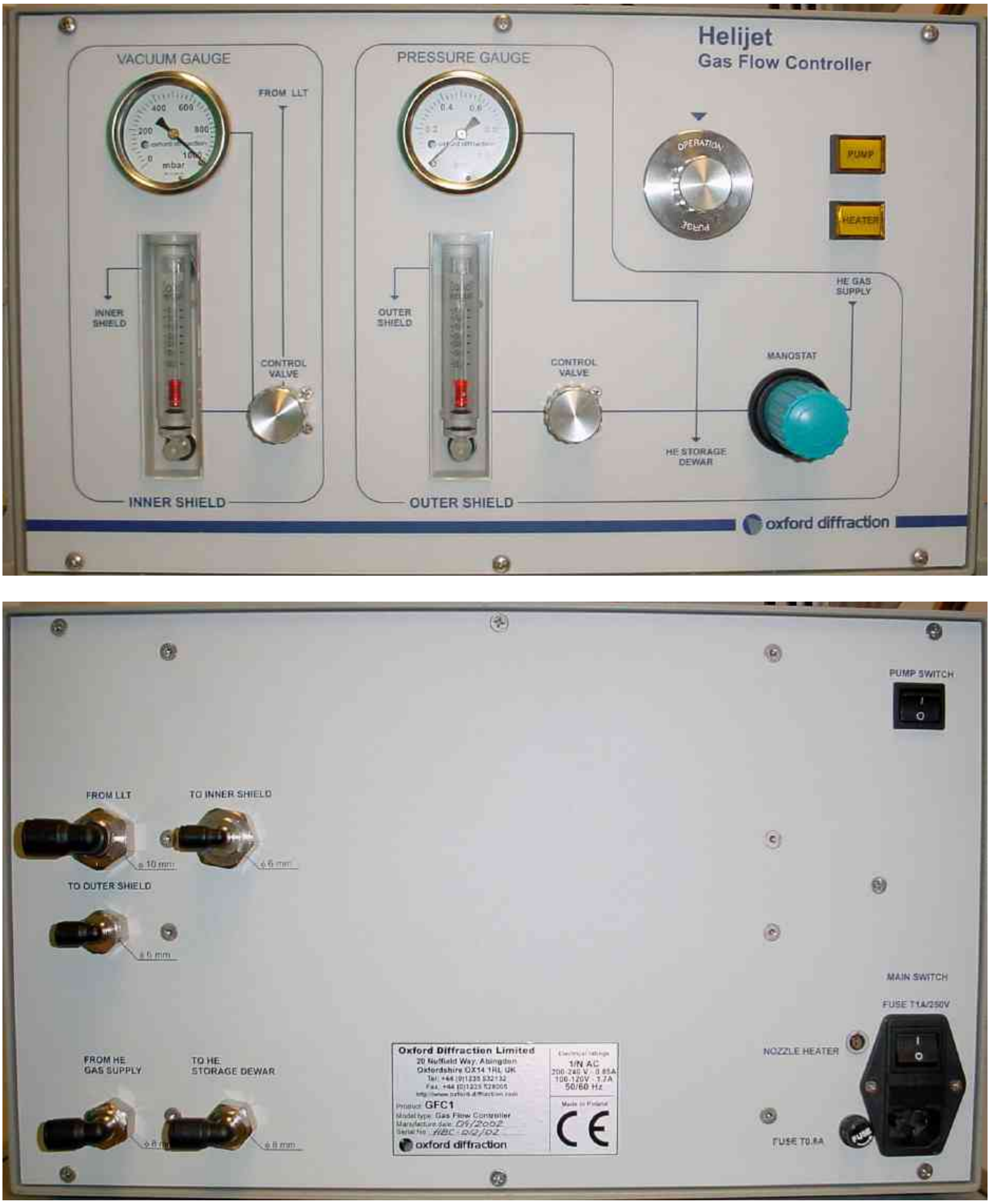


Figure 4-6 Gas Flow Controller (GFC) Front Panel (Top) and Rear Panel (Bottom)

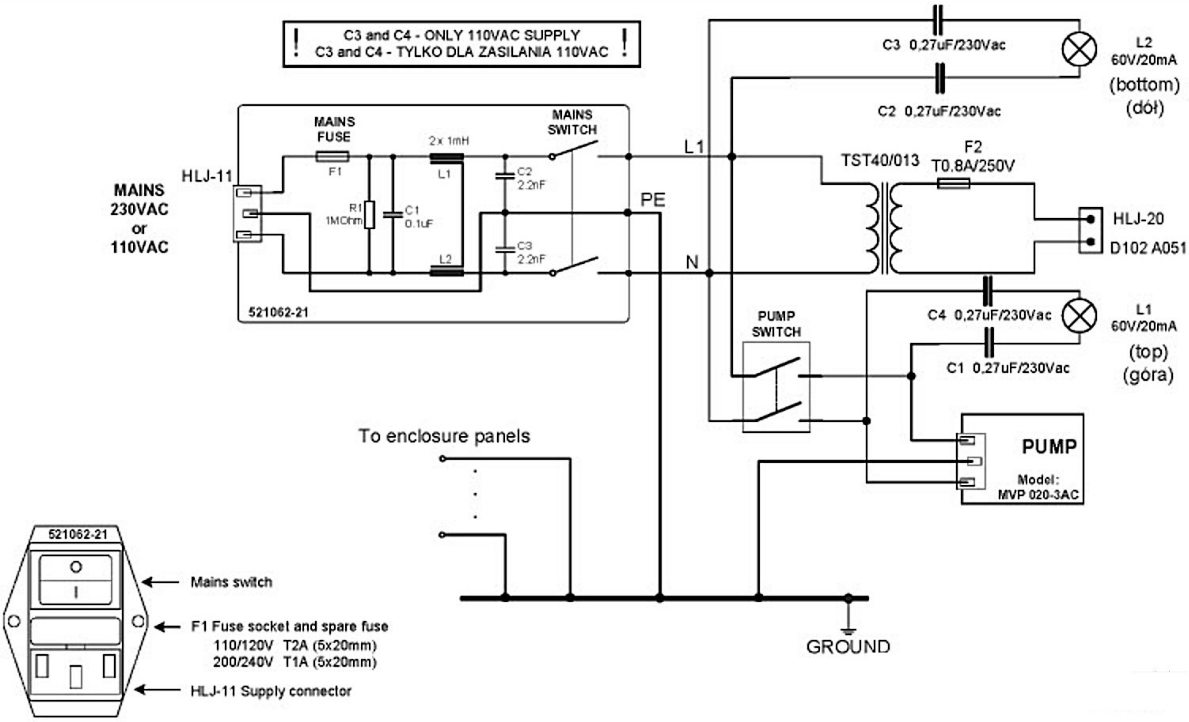


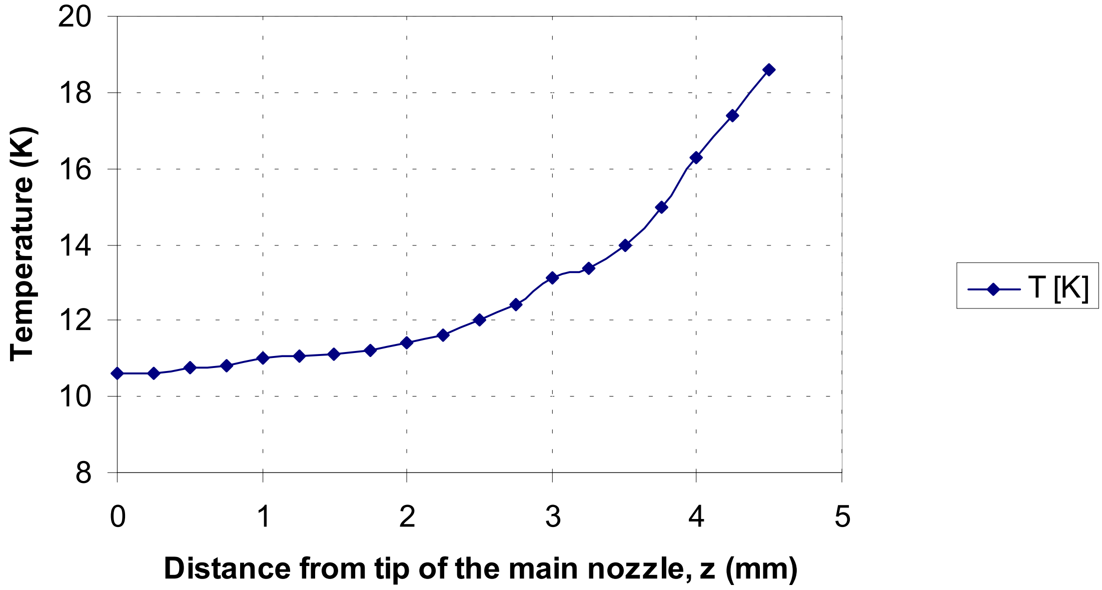
Figure 4-7 Schematic Diagram of the GFC1 Unit

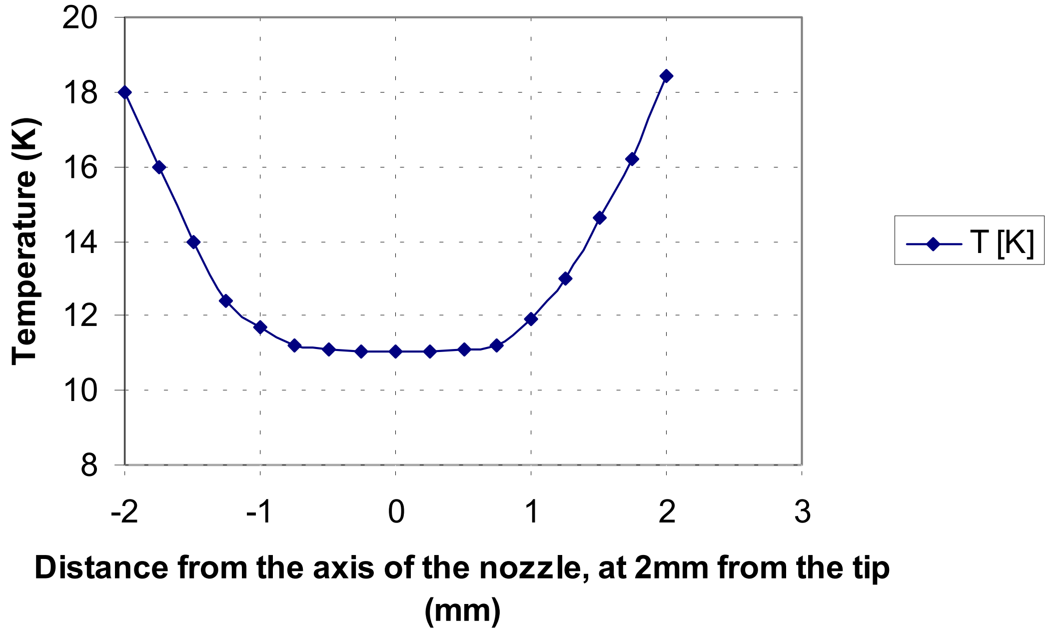
4.5. The Cryogen Transfer Tube

The LLT transfer tube is designed for ultra low loss performance. The cold exhaust gas from the cryostat flows along the tube, and the enthalpy of the gas is used to shield the flow of liquid from the room temperature surroundings. The LLT650 has an automated needle valve, which allows the flow rate to be optimised automatically. The LLT600 has a manual needle valve, the flow is set and the heater voltage is balanced against the required set temperature.

4.6. Operating Values

The graphs below show the typical temperature distribution of Helijet. The measurements were taken along and across the main stream. The helium consumption during the measurements was 2.1 litres per hour.





4.7. Safety Features

The Helijet head and LLT transfer tube have an overpressure valve located in the pump out port valve. These must not be covered or blocked as the consequences may be dangerous.

Vacuum spaces in cryogenic systems must have an overpressure relief valve for the following reasons:

- If the system is operated for an extended period and an air leak goes unnoticed. The air leaked into the vacuum space is likely to freeze onto the cold surfaces. Only when the system is warmed does it become apparent that a large volume has been collected. This can become dangerous as the as the air expands to fill the vacuum space above atmospheric pressure.

- If one of the vessels filled with helium liquid leaks fluid into the vacuum space this can cause the system to warm rapidly and fill the vacuum space to above atmospheric pressure.

5. Installation

5.1. Reception and Handling

5.1.1. Delivery

Perform the following steps on delivery of the system and before unpacking the equipment.

1. When the system arrives, check that there is no visible damage, with the delivery driver present. If damage has occurred contact the carrier and Oxford Diffraction **immediately**.

2. Check the number of delivered items. The total system is delivered in two separate boxes. One box contains the Gas Flow Controller (GFC1), the Helijet head, and the temperature controller (ITC502). The other box contains the transfer tube (LLT600). If any items are missing contact Oxford Diffraction within 3 days.

CAUTION
Do not remove the equipment from the packing crate until it has been moved to its designated installation site. The equipment has been carefully packed to protect the equipment from damage in transit. Removal of the packing equipment could make the equipment vulnerable to damage during transit.

3. Move the packing case into the designated installation site.

5.1.2. Unpacking

CAUTION
Before using the equipment, check that the voltage selector on the pump is set to the intended electrical supply voltage.

Checking the set voltage on the pump on the GFC1

1. Remove the lid from the cabinet by unscrewing four screws in the side panels.

2. Check that the voltage selector on the pump is set to the intended supply voltage.

3. Refit the lid and tighten all fixings. The GFC1 is now ready for assembly.

4. Check that the mains supply voltage indicated on the back of the GFC1 is correct for your local power supply.

Unpacking the Helijet head:

1. Remove the Helijet head carefully from its box.

2. Inspect the Helijet head for shipping damage.

3. The head must be evacuated with a suitable turbo-molecular pump (70l/sec) before operation to ensure that it works to optimum performance. Connect the pumping system to the NW16 flange on the side arm valve. The vacuum space is filled with an atmosphere of dry nitrogen for shipping.

4. Slowly open the valve anti-clockwise. The valve is fully open after 6 turns.

5. Leave the head pumping until the pressure reaches 1 x 10^{-5} mbar or preferably overnight.

6. Close the valve, vent the pumping station and remove the pumping line.

7. The head should now be ready for assembly.

Unpacking the ITC502:

1. Removed the ITC502 from its box.

2. Inspect the ITC502 for shipping damage.

3. Check the voltage selector located on the rear panel to ensure that it is set for the correct local voltage. The controller will be damaged if the instrument is powered up using the wrong voltage.

Unpacking the LLT transfer tube:

1. Carefully remove the LLT transfer tube from the packing box.

2. Inspect the LLT transfer tube for shipping damage. If there are any signs of damage, please report this immediately to the customer support department at Oxford Diffraction Ltd.

3. Evacuate the transfer tube to ensure that the insulating vacuum will be suitable for operation. This procedure is described on page 5 of the LLT manual.

5.1.3. Weights, Dimensions and Lifting Points

Description	Weight kg	Dimensions (width x height x depth) cm	Centre of gravity	Lifting points
Gas flow controller	12	45 x 26 x 38	Centre of box	Front panel handles
Helijet head	8	52 x 23 x 9	Head top plate	Side arm
Temperature controller	6.5	46 x 11 x 30	Right hand side	Front panel handles
LHe transfer tube	3	150 x 250 x 4	At 90 deg elbow	Support flexible section and at 90 deg elbow

All parts of the system can lifted into place by hand. No lifting equipment is required to assemble the system at ground level.

5.2. Preparation of Site and Services

5.2.1. Environmental Requirements

The ITC502 and GFC1 should be located not more that 2.5m from the head. They can either be free standing or will fit into a standard 19” rack with 8U height. Do not obstruct the side panels of the GFC1 as helium is vented through the side panels.

Existing Xcalibur users will require:

- About 2m of space to the left side of the diffractometer for the helium storage dewar during operation.
- At least 1.5m height above the top of the storage dewar to allow the LLT to be loaded and unloaded from the dewar.
- a mounting bracket (supplied) for the head. This will interface between the Xcalibur support arm and the top plate of the head.

Non-Xcalibur users should note that:

- the transfer tube flexible section is 2m long and therefore the location of the Helijet head must accommodate this.
- the Helijet head can be mounted in any orientation and the fixing points are shown on drawings HA-00-00-007-C, HA-00-00-008-C and HA09-00-001-C.
- the ceiling must be at least 1.5 m above the top of the storage dewar to allow the LLT to be loaded and unloaded from the dewar.

5.2.2. System Layout

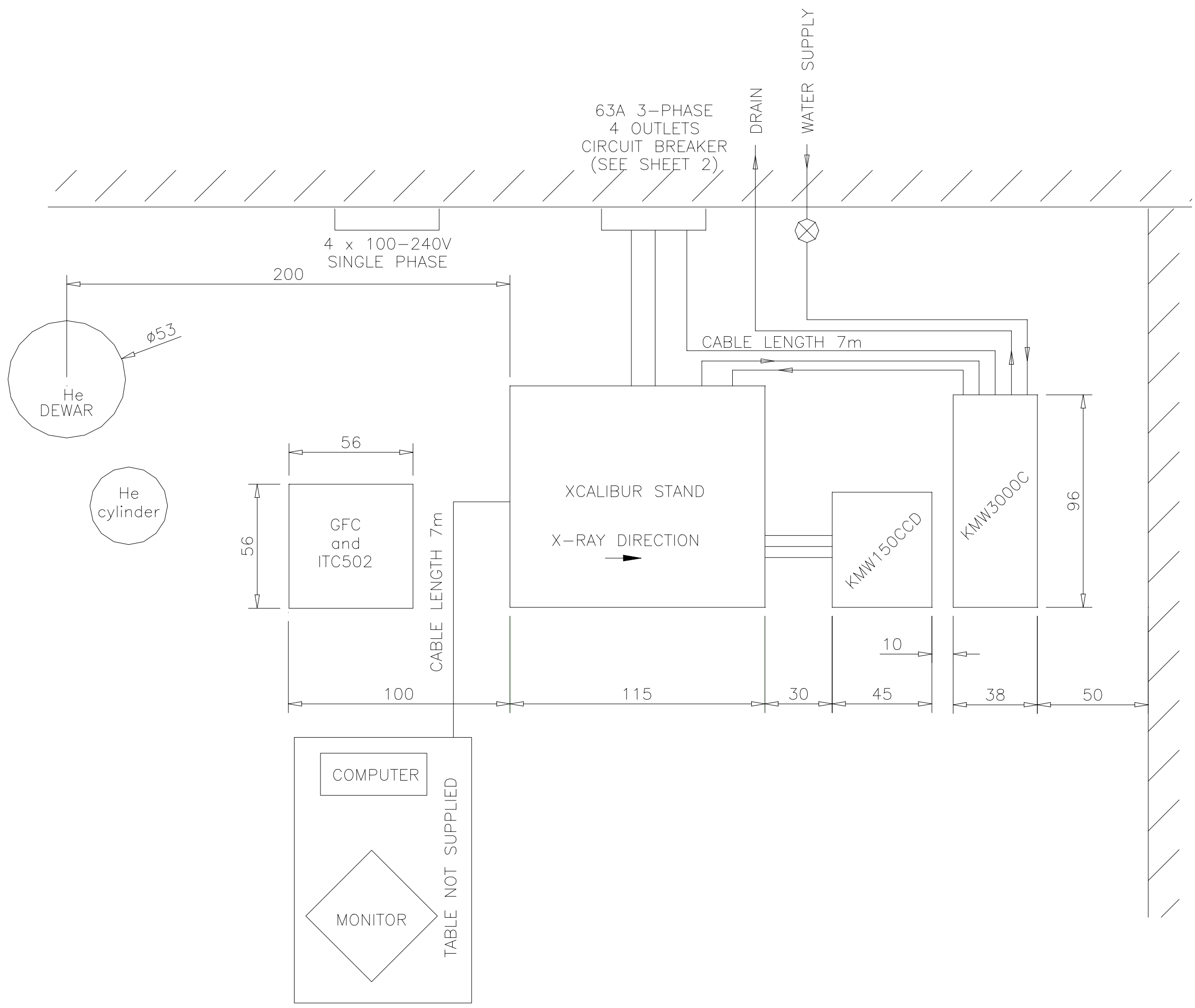


Figure 5-1 Site Layout

5.2.3. Electrical Services

The electrical supply must conform to the requirements stated in the SPECIFICATIONS section of this manual.

The system requires two single-phase outlets at the local voltage.

The system can be operated between 100 – 240V but the voltage selectors must be set to the intended supply voltage before operation.

The mains plug should be readily reachable (within 1.5 m) by the operator when the equipment has been installed.

Item	Voltage	Frequency	Current	Fuse type
ITC502	100 – 120V	50/60 Hz	1.6A	Type T (Slow blow)
ITC502	200 – 240V	50/60 Hz	0.8A	Type T (Slow blow)
GFC1	90 – 126V	50/60 Hz	1.7A	Type T
GFC1	180 – 254V	50/60 Hz	0.85A	Type T

5.3. Setting to Work

5.3.1. Equipment Required

The customer should supply the following equipment:

- 70l/sec Turbo pump and vacuum gauge
- 1.5M pumping line with KF16 vacuum fittings at 1 end
- KF16 clamp, carrier and o-ring
- 2 x 15/16” AF spanners
- 1 set of metric Allen keys
- protective gloves and mask
- Suitable helium storage dewar to fit 12 mm-diameter transfer tube (leg 130cm long) with access for 8mm Teflon tube to pressurise the storage dewar to a maximum of 1.5barg.
- Liquid helium
- Compressed helium gas cylinder (minimum of 100 bar cylinder) with suitable low-pressure regulator or a suitable supply of helium gas (recovery grade) delivering a minimum of 0.5 bar gauge and 30l/min flow, with gas regulator to fit onto 8mm Teflon tube supplied.
- dipstick for measuring the liquid helium level (optional)
- DVM (optional)
- hot air gun (optional)

5.3.2. Interfaces

A mounting plate (part numbers HA-00-00-007-C and -008-C) is supplied for fixing the Helijet head to Xcalibur.

If the Helijet head is to be mounted on any other apparatus then a suitable bracket must be made. The head can be mounted in any orientation but allow access to assemble the transfer tube into the side arm. Refer to drawing HA-09-00-001-C for details of the mounting points.

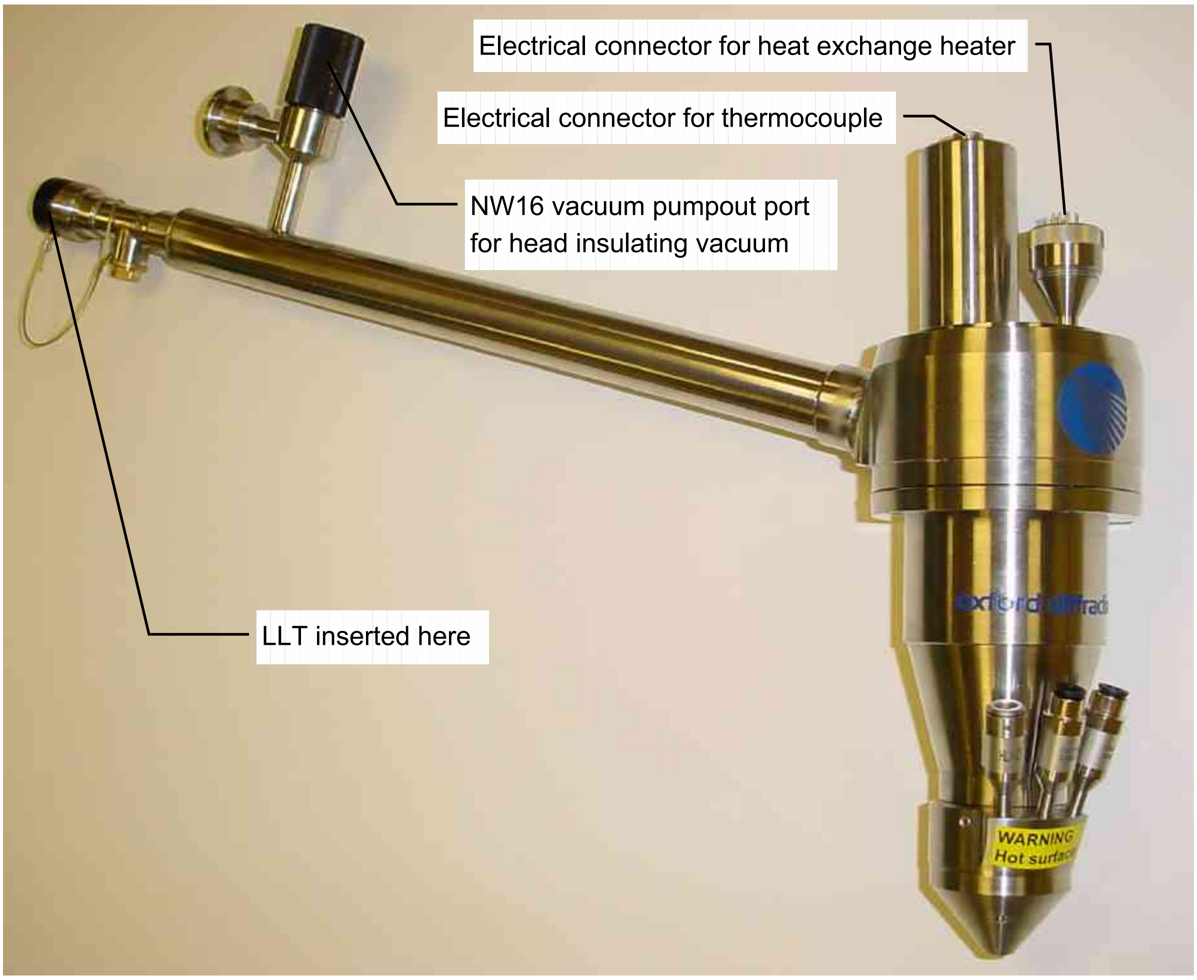


Figure 5-2 The Helijet Head

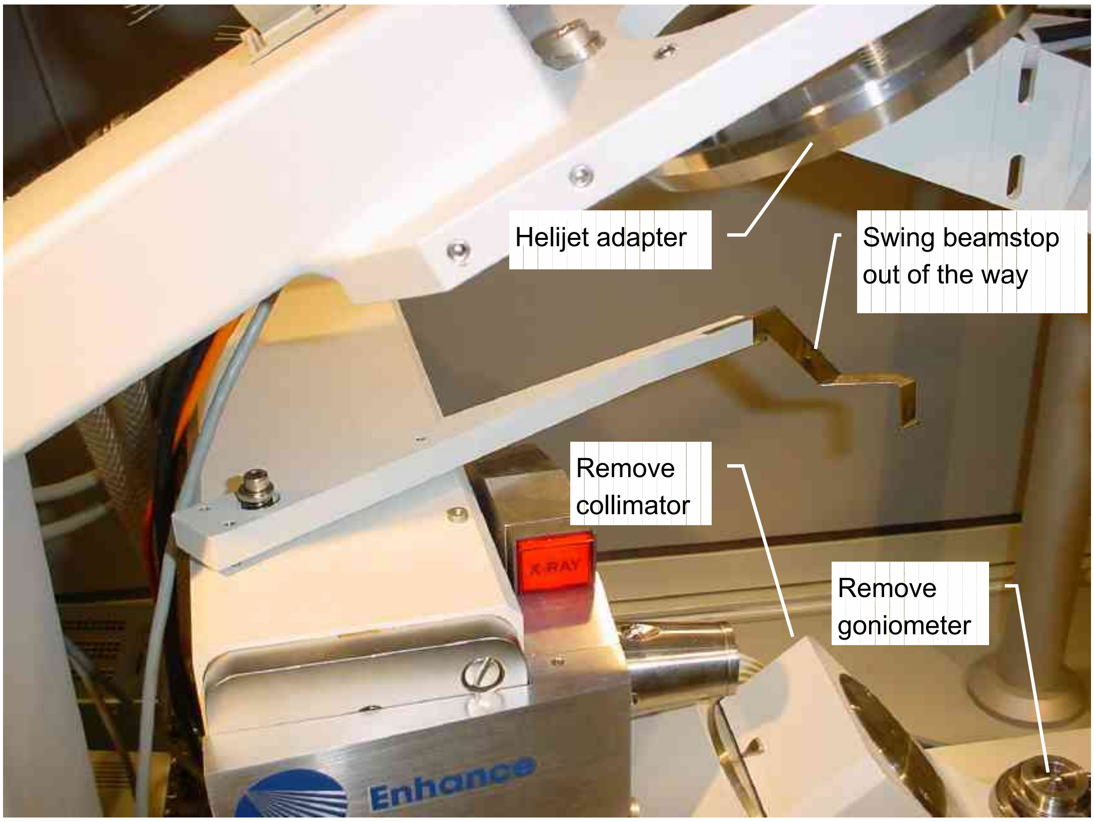
5.3.3. Mounting Helijet on Xcalibur

The following instructions show how to mount Helijet on the Xcalibur diffractometer. The instructions for mounting the Helijet system on other diffractometers will be different.

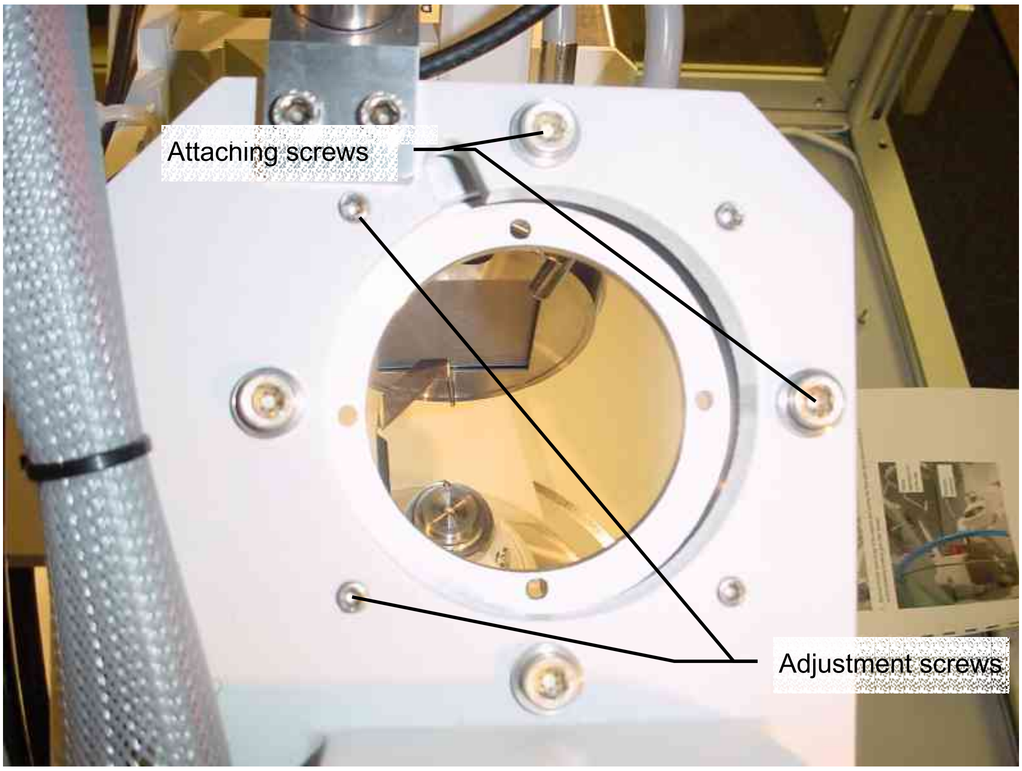
If in any doubt about how to mount the Helijet on your equipment, please contact Oxford Diffraction for further advice.

1. On the diffractometer dismount the goniometer head and collimator (if fitted).

2. Remove the top most screw of the support arm holding the fibre-optic lights and pivot the arm on the remaining screw to give clear access.

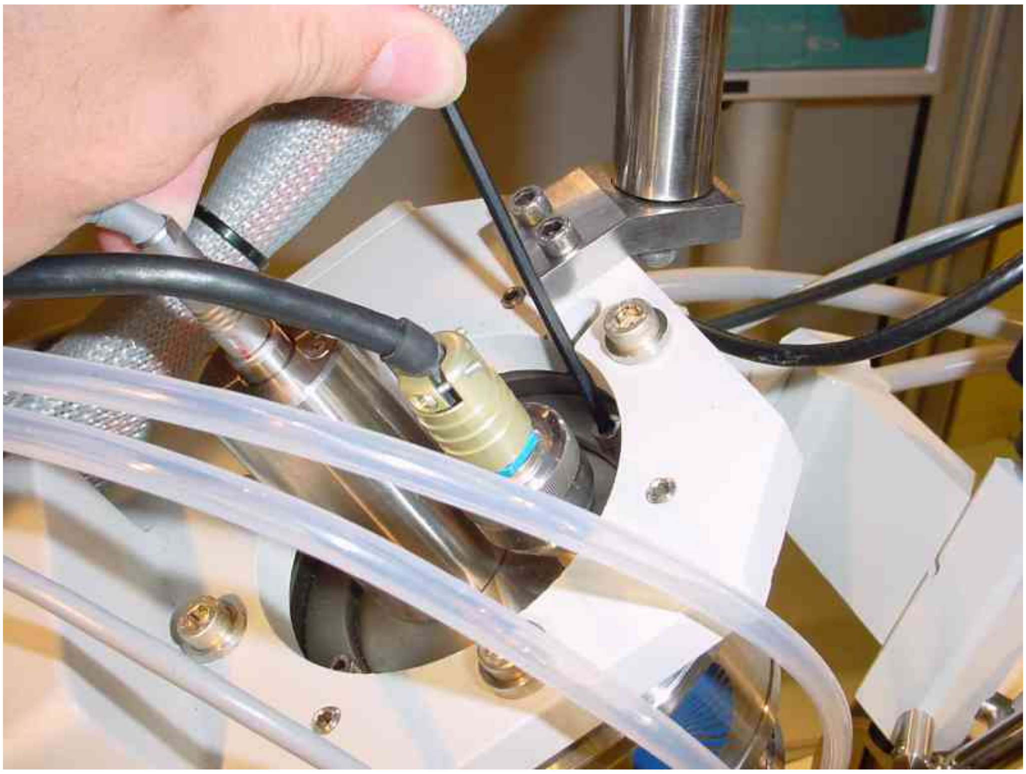


3. Attach Helijet adaptor to support arm using the 4 screws (with washers and springs) provided. Put in place the 4 adjustment screws.

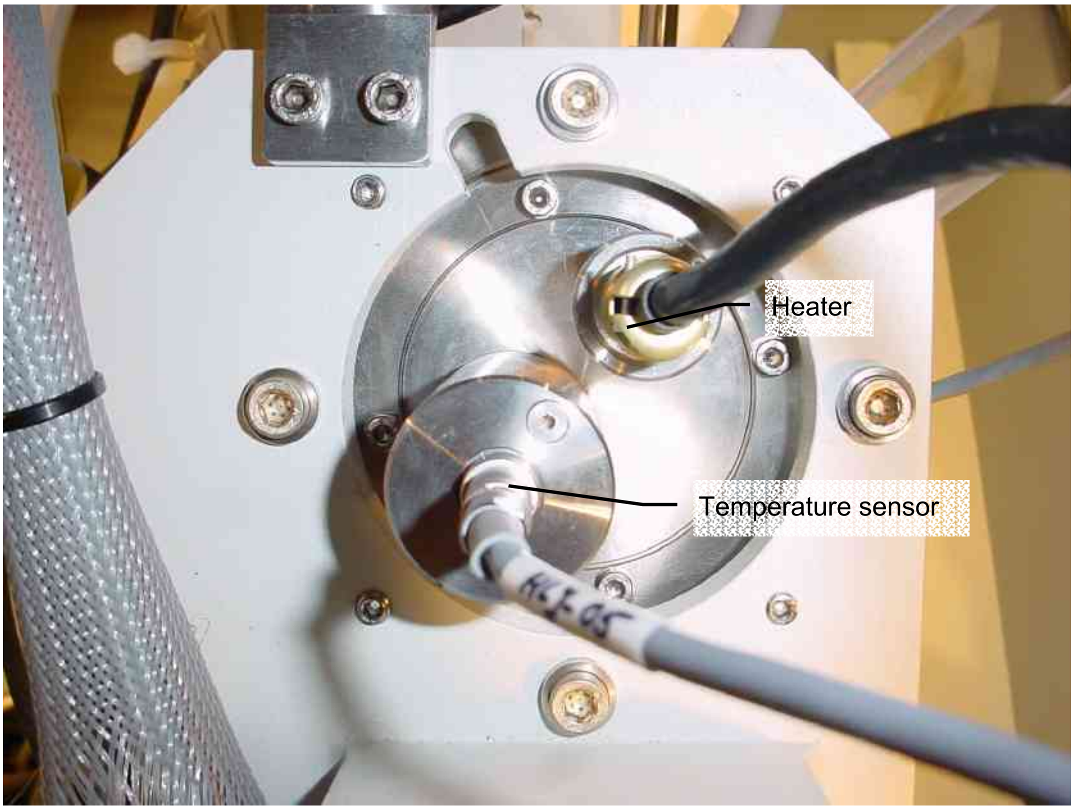


4. Insert the Helijet from the front of the Xcalibur and fit into the adaptor.

5. Secure the Helijet in place using the screws provided, whilst supporting from below.



6. Attach heater and temperature sensor cables to top of the Helijet head.

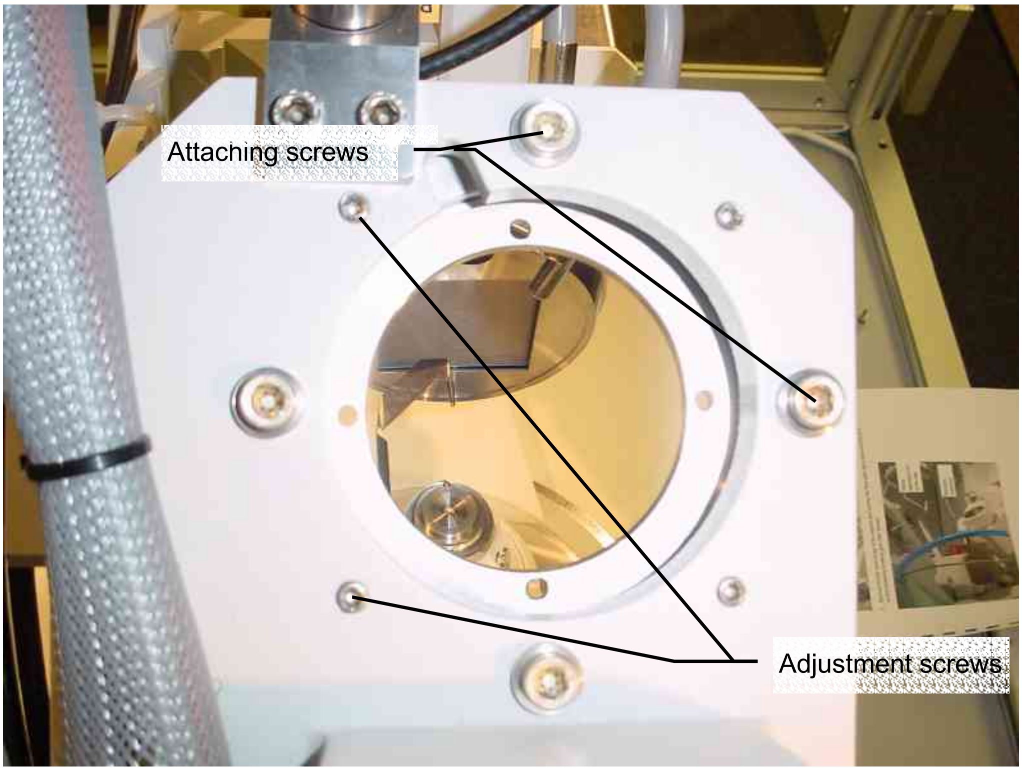


7. Attach the Teflon tubing to the Helijet nozzle.

8. Mount collimator and goniometer head.

5.3.4. Aligning Helijet on Xcalibur

1. Retract the adjustment screws in the support arm so that the support bracket is clamped against the underside of the support arm.



2. Place the 0.3mm alignment ball on the goniometer and visually check that the nozzle points at the alignment ball.

3. There should now be about an 8mm clearance from the end of the nozzle to the ball.

4. If the ball and the end of the nozzle are out of alignment then adjustment should be made using the 3 set screws in the base of the support arm. This is described in the Xcalibur manual.

5. Remove the alignment ball from the goniometer.

6. Now that rough alignment has been achieved the fine adjustment can be carried out using the adjustment and attaching screws in the support arm (see the illustration in step 1 of this procedure).

7. Place the alignment tip in the end of the Helijet nozzle.

8. Fit a bare glass fibre to the XYZ head and place on the goniometer.

9. Align optically the end of the fibre with the centre of the goniometer axis.

10. Adjust the screws until the alignment tip nearly comes in contact with the end of the fibre.

11. Ensure all screws are tight.

12. Remove the goniometer head.

13. Remove the alignment tip.

14. Alignment is complete when the end of the nozzle is 3mm away from the sample.

5.4. Storage

Before installation commences, or when the equipment is not being used for extended periods, store the Helijet in accordance with the environmental conditions for temperature and humidity stated in the SPECIFICATIONS section of this manual.

To store the Helium transfer tube (LLT) it is recommended that the flexible section is supported horizontally with the dewar leg suspended vertically downwards. This can be done by fixing 3 angled brackets to a suitable wall for support of the flexible section.

It is recommended that the Helijet head remains mounted on the diffractometer. If for any reason the head is removed, store it in the packing box supplied.

The ITC502 and GFC1 are supplied as freestanding units but can be mounted into a 19” rack if the rack mounting option has been purchased.

6. Operating Instructions

6.1. Warnings and Cautions

WARNINGS

1. **Read and understand the safety notices in the Health and Safety Information chapter of this manual before performing any of the operating procedures detailed in this chapter.**
2. **Do not touch the nozzle of the Helijet head when it is operating, as it gets hot and could cause burns. An indicator light on the GFC shows when the heater is operating.**

Helijet is supplied as a complete system. The temperature controller and head have therefore been set up in the factory to prevent the maximum safe operating temperature of the head being exceeded and to limit the maximum heater voltage to a safe level.

CAUTION
Ensure that the maximum heater voltage and operating temperature of the head are limited to safe levels in the temperature controller. If the maximum safe operating levels of these parameters are exceeded it is possible to cause serious damage to the equipment.

If you use a different temperature controller you must limit the maximum heater voltage, and operating temperature of the head, to a safe level. Refer to the Specifications section of this manual for the recommended values of the 'Heater Voltage Limit' and the 'Temperature Limit (T_{HOT})'. Refer to the information supplied with the temperature controller for information about how to set these limits correctly.

You will be unable to use a power supply or controller made by any other manufacturer other than Oxford Instruments ITC range.

6.2. Controls and Indicators

6.2.1. ITC502 Front Panel Controls

POWER	The main ON/OFF switch. A green lamp illuminates whenever the instrument is switched on.
ADJUST	The red RAISE and LOWER buttons provide the main means of adjusting any parameter. They have no effect on their own and are always used together with one of the other buttons. Whenever a parameter is being adjusted, its current value is shown on the main display. Setting a value involves pressing RAISE and/or LOWER until the required value is shown. Operation of the RAISE and LOWER controls has been designed to allow large changes to be made relatively quickly whilst at the same time enabling any

value to be set exactly. Pressing RAISE or LOWER briefly will cause the value to change by one unit. If the button is held in, the last figure will start to change at about 5 units per second. After 2 seconds, an approximately 10-fold increase in rate will occur, followed after further 2 second intervals by two more rate increases. Altogether there are 4 different rates. Whenever RAISE or LOWER is released, the next lower speed will be selected. This allows the user to "home-in" on the required value most ergonomically.

A secondary use of RAISE and LOWER is in conjunction with LOC/REM, to enter the TEST & CONFIGURATION mode, as described below.

CONTROLS

Control of the instrument may either be LOCAL from the front panel or REMOTE via the RS232 interface. The LOC/REM button may be used to switch between LOCAL and REMOTE.

When LOCK is lit, the instrument is locked into either local or remote control and the LOC/REM button has no effect. At power up, ITC502 is locked in LOCAL, since at that time the instrument has no way of "knowing" if there is a computer connected to the RS232 interface.

When ITC502 is in REMOTE, many of the front panel controls are disabled. Those controls, which only affect the display, will still work but those, which could change the operation of the instrument, will not. If LOCK is lit whilst in REMOTE, all the front panel controls are inoperative.

The LOC/REM control button has a number of secondary SELECT functions which are obtained by pressing this button whilst one or more other buttons are held depressed. If LOC/REM is pressed whilst both RAISE and LOWER are held in, ITC502 enters the TEST mode (described elsewhere). If LOC/REM is pressed whilst the LIMIT button is held in, current calibration and configuration data is STORED in the non-volatile memory and so is retained at power-up.

HEATER

The normal way in which ITC502 effects its control is by applying power to a heater. In MANUAL control the heater voltage may be varied by RAISE and LOWER. In AUTOMATIC control the heater voltage is varied in response to the difference between a measured temperature and a set point.

Pressing AUTO or MAN switches between the MANUAL and AUTOMATIC states. In either case, whilst the button is pressed the main display gives an approximate indication of the output voltage. (N.B. This is not a calibrated parameter. If a particular requirement needs an accurate value for the heater voltage, a meter should be connected to the heater leads). When ITC502 is in REMOTE control, switching between AUTO and MANUAL is disabled, but it is still possible to use the buttons to display the output voltage, unless ITC502 is in LOCK & REMOTE.

A 10-segment bar graph is provided to give a continuous indication of heater operation.

When the maximum heater voltage has been limited (see ITC502 manual section 5), the main display will indicate the actual output voltage whilst AUTO

	or MAN are pressed. The bar-graph display will automatically be scaled so that an output on the limit will light all 10 bars.
GAS FLOW	Two buttons, with associated lamps, allow AUTOMATIC or MANUAL control of gas flow to be selected. These are provided for use with the LLT. They may be used to vary the flow of the helium in the same way that the heater controls are used to vary the heating power. When the auto needle valve cable is connected, the GAS MAN lamp will flash for a time after switch-on, whilst the controller establishes a reference position for the needle valve. During this time the Gas Flow Buttons will be disabled.
SWEEP	The ITC502 incorporates a programmable sweep facility. This is controlled by a single RUN/PROGRAM button with three lamps. It is described in detail in section 6 of the ITC502 manual.
DISPLAY	The main display normally indicates the measured temperature. Where more than one input channel is fitted, the SENSOR button may be used to switch between input sensors. This affects only the displayed temperature, and so remains operational even in REMOTE control. Whilst the button is pressed, the display indicates the range code for the sensor being selected. This serves as a reminder of which sensor has been associated with which channel. Pressing the SET button switches the display to indicate the set temperature. Provided the controller is in LOCAL, the RAISE and LOWER buttons may be used to adjust the set point. Similarly, pressing the PROP, INT and DERIV buttons allows the value of the corresponding control terms to be displayed and modified. The display block includes two additional buttons LIMIT and CAL. These are rarely used controls and are recessed behind the front panel to prevent inadvertent operation. They may be operated using a pointed object, such as the point of a pencil. Their operation is described in the ITC502 manual, sections 5 & 7 respectively.

Switch on the instrument by pressing the POWER switch. Check that the green POWER lamp lights.

After about one second the display will show "S" followed by a number, which indicates the instrument's "ISOBUS" address (see below). Alternatively if the instrument is fitted with an optional GPIB card the display will show "G" followed by a number, indicating its GPIB address assuming this has been selected. In either case, this will be followed by the word "PASS". This indicates that the ITC502 has completed its self-test and initialisation.

After a further pause the internal SAFETY RELAY will close. This links the heater to the controller output. At the same time, the display will show the measured temperature for Channel 1.

If the auto needle valve cable is connected, the GAS MAN lamp may continue to flash for several minutes whilst the needle valve is reset. During this period, Gas Flow control is disabled, but all other functions operate normally.

The ITC502 will now be under LOCAL control from the front panel, with the HEATER and GAS FLOW in MANUAL and the heater voltage at zero.

6.2.2. GFC1

- The helium gas supply can be switched between the outer and inner shield using the 3-way bypass valve.
- The on/off switches are located on the back panel of the GFC. The lower mains switch switches on power to the unit and also turns on the heater. The upper switch turns on the internal diaphragm pump. The lower switch must be on to operate the pump.
- Readings from the flowmeters on the front panel are taken from the top of the floats.
- The inner shield flow is controlled by the manual control valve situated on the left-hand side on the front panel of the GFC1 unit.
- The outer shield flow is controlled by the manual control valve located on the right hand side on the front panel of the GFC1 unit.
- Pressure in the storage dewar is controlled by the blue manostat valve.

6.3. Helium Requirements

A standard helium storage dewar cannot be refilled during operation. It is therefore important to make an estimate of the duration of the experiment, and thus an estimate of the amount of liquid helium required to complete the experiment before starting to operate the Helijet system. Then ensure that the helium dewar contains sufficient liquid helium.

To calculate the liquid helium required for an experiment:

Estimated volume of liquid helium required (in litres) = (length of experiment in hours x 2) + 6

For example:
An experiment lasting one hour requires 8 litres of liquid helium
An experiment lasting two hours requires 10 litres of liquid helium.

This calculation gives an estimate only of the helium requirements. The actual helium requirements for a particular experiment depend on specific experimental conditions. If in any doubt about helium requirements ensure that at the start of the experiment the dewar contains more helium than the estimated amount.

6.4. Set Up and Assembly

To prevent ice forming on the goniometer head it is recommended that a heated head is used.

1. Place the nozzle as close to the sample as possible to prevent icing. The limitation on this spacing depends on the diffraction angle required and the angle between the nozzle and the incoming x-ray beam. In almost every case the nozzle should be within 5 mm of the sample.

2. It is recommended that the head is installed at an angle of >35° to the horizontal plane.

3. Centre the jet on the sample as accurately as possible.
   This can be done by first adjusting the nozzle so that the sample is in the plane of the nozzle, then using the adjustment screws on the mounting plate for the fine alignment.

   Another method is to use the Oxford Diffraction alignment tip. Slide the device into the nozzle. Place a sample mount with no sample on the goniometer. Adjust the nozzle so that the sharp tip is almost touching the place where the sample would be (Xcalibur users can align the tip using the video monitor. Remove the sample mount. Remove the alignment tip, and then place the sample on the goniometer.

6.5. Switch-on Procedure

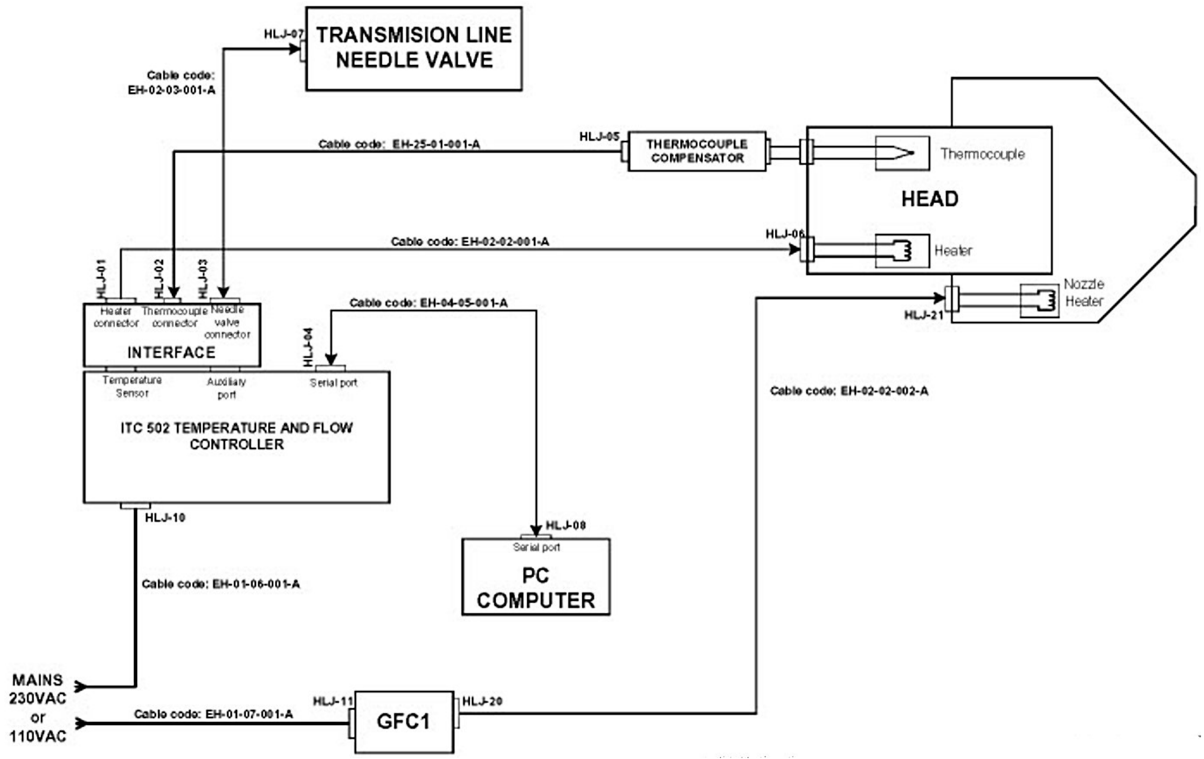


Figure 6-1 Helijet Electrical Connections

1. Ensure that the ITC temperature controller is connected correctly (see Figure 6-1 Helijet Electrical Connections).

Cable	Connection on temperature controller interface
Helijet head cable (EH-02-02-001-A)	heater connector socket
Thermocouple compensatory cable (EH-25-01-001-A)	thermocouple connector socket
Auto needle valve cable (EH-02-03-001-A)	needle valve connector socket

2. Connect the temperature controller to the mains.

3. Switch on the temperature controller.

4. Press the SENSOR button until the Sensor 1 LED is lit. The main display should show the code for the sensor fitted to the cryostat. Refer to the Specification section of this manual for the correct code. The calibration in the temperature controller has been set up for the thermometer in the Helijet head. If the code displayed is incorrect, please refer to the temperature controller manual.
The thermocouple reference junction is electronically controlled by the compensatory attached to the head.

5. Check that room temperature is indicated on the display. If not, refer to the details of the cryostat wiring and check for faults.

6. Switch off the temperature controller and then plug the auto needle valve cable (EH-02-03-001-A) into the flying lead from the needle valve body, if supplied.

7. Switch on the temperature controller. The LED on the gas flow panel will flash for up to 4 minutes 30 seconds as the temperature controller 'initialises' the auto needle valve. This process will stop as soon as the valve is fully closed. The buttons on the gas flow panel are disabled during initialisation, but the other features of the temperature controller can be used in the normal way. When the LED stops flashing the initialisation process is complete.
Note: If the auto needle valve is unplugged during initialisation the process will stop. It will not start again even if the auto needle valve is reconnected. Switch the temperature controller off and on again to re-start initialisation.

8. On the GFC1 set the control and manostat valves to the closed position (that is, for the control valve turn the valve control knobs fully clockwise, for the manostat valve turn anti-clockwise to close). Set the purge/operation knob to operation. Then on the back of the GFC1 switch on both power switches.

6.5.1. Computer Control

Although the Helijet does not require a computer, but it can be controlled from a computer using the RS232 interface, which is always provided, or the optional GPIB (IEEE-488) interface. In either case, you can use the Oxford Instruments ObjectBench version 2.8.7 software provided. Microsoft™ Windows® 95 or 98 is required.

Connect the ITC502 to a PC-compatible computer using a standard RS232 cable (not supplied).

Details are as follows:
Connect the computer via a 25 way D-socket on the rear panel of the temperature controller. The controller is configured as a DCE with the standard pin outs given below. Most computer RS232 interfaces are configured as a DTE and are fitted with a 25 way D plug. For this type of connector, a simple lead connecting pin 1 to pin 1, pin 2 to pin 2 and so on is all that is required. For computers fitted with a 9 way D plug for RS232, (AT style COM port), a standard "AT lead" fitted with a 9 way socket and a 25 way plug is required.

If you plug the computer into a different electricity supply circuit from the controller, you must use an Oxford Instruments ISOBUS cable to ensure electrical isolation. The optional GPIB (IEEE-488) interface can be supplied complete with cables and connectors for controlling the temperature controller from a computer.

6.6. Operating Procedures

6.6.1. Assembling the Transfer Tube

Use this procedure to assemble the transfer and also if:

- you have problems running the system
- you need to check and adjust the lock nut on a transfer tube that has been supplied as part of a system
- the transfer tube has to be adjusted to fit a different Helijet.

Equipment required: 15/16" or 24mm spanner

The nut and lock nuts are used to compress a collet, so that the assembly is firmly fixed to the transfer tube arm. The components are labelled in Figure 6-2.

1. Hold the nut and loosen the lock nut using two spanners so that the assembly can slide freely along the transfer tube delivery arm.
2. Push the transfer tube delivery arm fully into the Helijet side arm, so that the PTFE seal meets the mating surface inside the side arm. (Push the transfer tube in as far as it will go.)
3. Screw the knurled nut fully onto the thread on the entry arm, and then unscrew it by two or three turns.
4. Gently push the transfer tube delivery arm into the side arm to make sure that the PTFE seal is seating while you tighten the lock nut onto the transfer tube again.

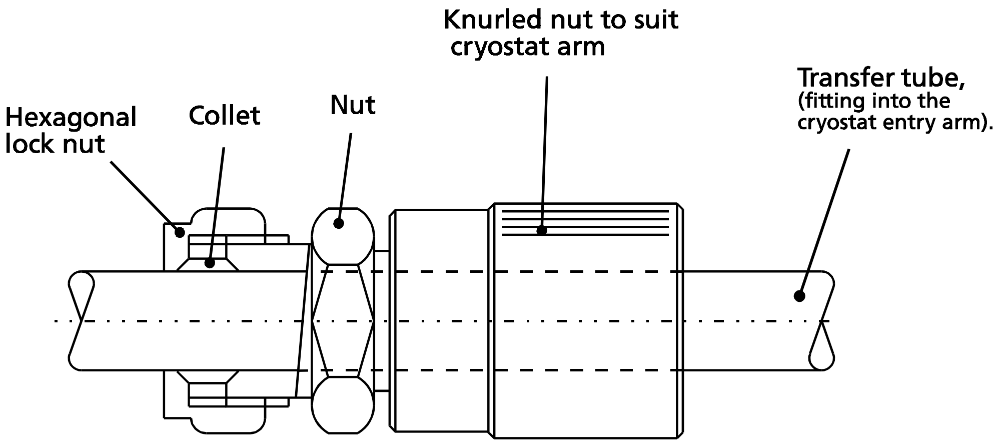


Figure 6-2 Transfer Tube Lock Nut

Once the transfer tube lock nut has been set in place, check it periodically to ensure that the locking collet has not moved due to the collet slipping on the transfer tube or over tightening on the knurled nut.

Ensure that all electrical connections are made as described in Figure 6-1 Helijet Electrical Connections and that the power is on to both the ITC and the GFC1. Ensure that all Teflon tubes

are connected in the correct order as described in the diagram below except for the 10mm return line from the LLT.

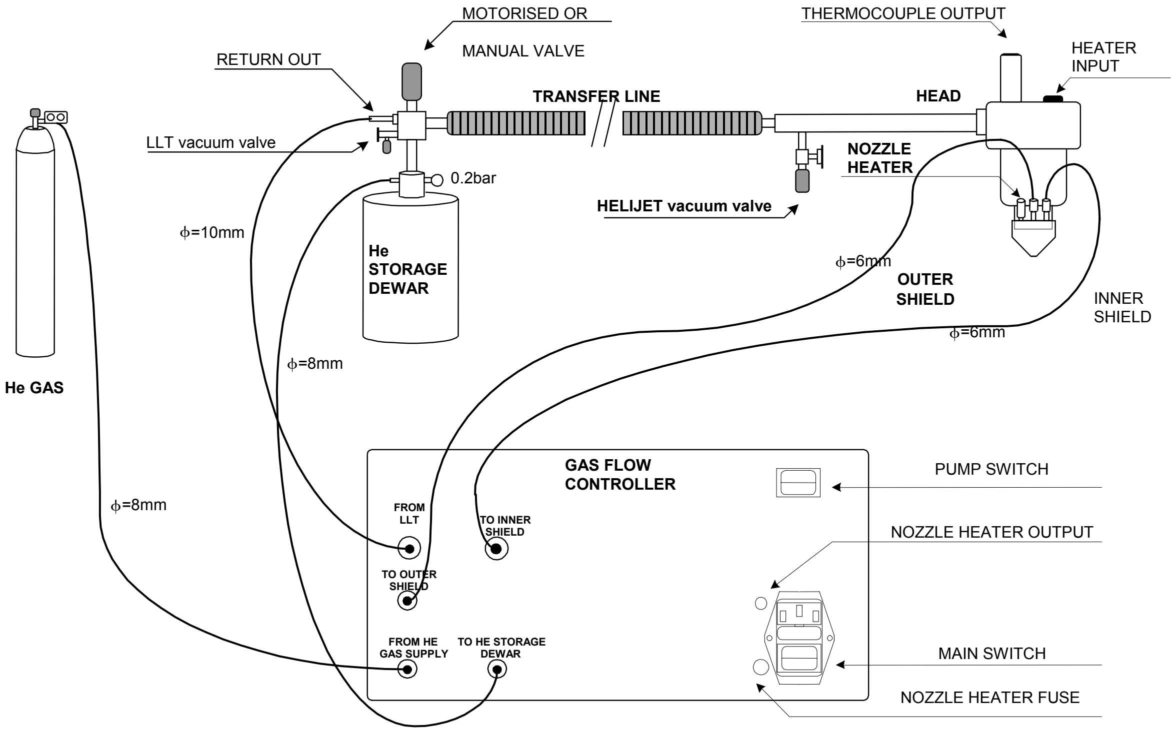


Figure 6-3 Assembly of Complete System

6.6.2. Cooling Down

1. Ensure that all tubes are connected to the correct ports as described in figure 6-3 except for the 10mm tube, this should be connected after the transfer tube has been inserted into the storage dewar.

2. Close the three control valves and put the 3-way valve into operation position, (located on front panel of GFC1).

3. Switch on the ITC and GFC1. Ensure that the installation procedure has been followed and the correct mains voltages have been set.

4. Ensure that all electrical plugs are connected and that the ITC is giving a room temperature reading as described in section 6-5.

5. Turn on the nozzle heater, (bottom switch located on back of GFC1).

6. Turn on the pump, (top switch located on back of GFC1).

7. The temperature reading on the ITC should rise above 300K; this is an indication that the nozzle heater is working correctly.

8. Open the needle valve on the transfer tube 4 turns or if the auto needle valve has been supplied open to 100%.

9. Slowly insert the 12mm diameter leg of transfer tube into the helium storage dewar, ensuring that the pressure in the dewar does not exceed 0.5bar, open the storage dewar vent if required. Some liquid will be lost due to the cooling of the transfer tube. If you try to cool the leg too quickly a large amount of liquid will be wasted, be careful that the exhaust gas does not come in contact with skin as this will burn.

10. Make sure that the 10mm tube from the GFC1 is connected to the return line on the transfer tube.

11. Make sure that the 8mm tube from the GFC1 is connected to the vent valve on the storage dewar.

12. Set the pressure on the helium gas supply to 1.5 bar.

13. Slowly open the blue manostat valve located in the bottom right hand corner of the GFC1 front panel and set the pressure on the outer shield pressure gauge to 0.3 bar.

14. Turn the 3-way valve to purge and set the flow on the outer shield flow gauge to 1.5 l/hr and leave for 2 – 3 minutes, this will purge the GFC1.

15. Ensure that the vent valve on storage dewar is open.

16. Remove the protective cover from the end of the transfer tube and check that the white Teflon seal is clean, dry and free from grease. Ensure that there is no damage to the seal faces and it is in the correct location.

17. Check that there is a gas flow from the end of the transfer tube; placing the end of the transfer tube into a small amount of alcohol and looking for bubbles can check this.

18. When you are happy that there is a flow through the transfer tube and it has been loaded into the storage dewar, you should insert the transfer tube into the Helijet head sidearm and engage the locknut one turn.

19. Tighten the lock nut hand tight. This allows the helium to follow the correct path through the head. This will need to be checked periodically during cool down as the internal components can cool at different rates therefore causing differential contracting.

20. Turn the 3-way valve to operation and set the flow on the outer shield flow gauge to 0.7 l/hr and leave for 2 – 3 minutes. This will purge the outer nozzle.

21. After about 8 – 10 minutes the temperature should start to drop.

22. When the temperature reaches about 100K open the inner flow control valve and set the pressure to 200 mbar. This should set the inner shield flow to about 0.4 l/hr.

23. The temperature should reach 15 K within 10 – 12 minutes from start.

24. Check that the nozzle is free from ice before proceeding. Ice may form due to moisture in the head if the system has previously been shut down incorrectly. The ice can be removed using a small metal object or paint brush to knock off the crystals but you should not insert this into the central tube, as this will damage the thermocouple. The ice should diminish if the system has been assembled, and is working, correctly.

25. When the base temperature of about 10 K is achieved, reduce the dewar pressure to 0.2 bar using the manostat valve.

26. Re-adjust the outer shield flow to 0.7 l/hr.

27. The head should now be operational.

6.6.3. Flash Cooling the Crystal

It is best practise to flash cool the crystal as crystals are often suspended in either high viscosity oil or grease. The following procedure is for flash cooling the crystal.

WARNING
Wear suitable protective gloves when performing this procedure to avoid injury caused by cold burns.

1. Set the temperature of the ITC502 to 75 K and wait for the temperature to stabilise. The ITC502 operating procedure can be found in section 4 of the operator's handbook.

2. When the temperature has stabilised, carefully place the crystal into the gas flow to flash freeze the crystal and liquid suspension.

3. Screw the goniometer head in to place and monitor the crystal using the video monitor to check for ice crystals.

4. You should now be ready to start testing.

6.6.4. Stabilising the Temperature

To improve the stability you can adjust the PID settings; the following procedure describes how to do this.

The PROPORTIONAL, INTEGRAL and DERIVATIVE control terms may be displayed and set by means of the PROP, INT and DERIV buttons. (Refer to section 14 of the ITC502 manual for a definition of the three control terms).

PROP indicates the PROPORTIONAL BAND as a percentage of the input span, covering a range of 0 to 199.9% in steps of 0.1%.

INT indicates the INTEGRAL ACTION TIME in minutes, covering a range of 0 to 140 minutes in steps of 0.1 minute.

DERIV indicates the DERIVATIVE ACTION TIME in minutes, covering a range of 0 to 273 minutes (Though values beyond 70 minutes are unlikely to be required in practice.).

RAISE and LOWER may be used to vary the control terms whilst in LOCAL control. Once a set of values has been chosen, they may be retained in the ITC502 's non-volatile memory and will not need resetting at switch on, provided a STORE operation is performed (section5.4 of ITC502 manual).

The P, I & D terms cover a large range of values to cover systems ranging from a small laboratory cryostat to a large furnace. For most laboratory applications a PROPORTIONAL BAND of 2 to 20% is appropriate, with an INTEGRAL TIME of 1 to 10 minutes.

The main purpose of DERIVATIVE action is to reduce overshoot, when approaching a new set temperature. For many small systems derivative action will not be required and may be left at zero. (Hold LOWER pressed for a second after 000.0 is displayed to ensure that there is not a small residual setting of less than 0.05 minute which will show as zero).

The PROP and INT controls should not normally be set to zero, since this would correspond to ON/OFF control (Section 14 of the ITC502 manual).

Section 14 of the ITC502 manual covers the theory behind P, I, D control. The following procedure gives a good guide to setting the controls to a value that is close to optimum.

1. Set INT for a time much longer than the expected response time of the system.

2. Set DERIV to zero.

3. Select AUTO and reduce PROP until the temperature starts to oscillate above and below some mean value (not necessarily the set point).

4. Time the period of oscillation (in minutes). This is a measure of the response time of the system.

5. Set INT to a value approximately equal to the response time. Then increase the PROP setting to a point where oscillation just ceases. Note the value of PROP at this point, and then set it to approximately double this value. This gives a good starting point for THE PROP and INT control terms.

6. Test how the system responds to step changes in the SET point and modify the PROP and INT settings for a reasonably fast response without excessive overshoot.

7. If overshoot remains a problem following a large step change in SET, try the effect of adding some DERIVATIVE action. A good initial setting is half to one third of the system response time measured above. This will probably require PROP to be re-optimised for best results.

8. When optimising P, I and D the aim should be to achieve the lowest values of all three terms, consistent with no oscillation and an acceptably small amount of overshoot. This will give the fastest response for the system.

When adjusting the control terms remember that **reducing** the PROPORTIONAL BAND **increases** the controller gain. This can cause confusion when first encountering the concept of PID control.

Standard PID table for Helijet operation using the ITC502

Temperature range	**P**	**I**	**D**
15 – 70K	2.8	0.7	0
70.1 – 200K	2.8	0.7	0
200.1 – 350K	2.8	0.7	0

With an inner shield flow setting of 200 mbar, the operating temperature range is between 15 – 75K.

To set a temperature, press and hold the set button then adjust the set point by using the raise or lower buttons.

To go to the set temperature press the auto button on the heater section of the ITC front panel.

The temperature should become stable within about 5 minutes to +/- 0.2 K but the time will depend upon the temperature jump.

If you require to work above 75 K it is possible to increase the upper limit to about 100 K by reducing the pressure on the inner shield flow to 125 mbar and reducing the pressure in the storage dewar to 0.12 bar using the blue manostat valve.

You should not go lower than 0.12 bar in the storage dewar because that would cause the cold stream to stall and it would then be possible for moisture to form inside the head.

6.6.5. Shutting Down

1. Set the temperature to 280 K and ensure that the ITC is in auto mode (this will turn the heater to a maximum output of 40.0V and also regulate the heater power as the system warms).
2. Close the inner shield valve, this helps warm the system as it stops the counter flow cooling along the transfer tube.
3. Close the needle valve located on the transfer tube 3 ½ turns so that it remains ½ a turn open.
4. Reduce the dewar pressure to 0.05 bar.
5. Turn off the pump in the GFC1.
6. Vent the pump to an atmosphere of helium by using the operation/purge valve in the purge position.
7. When the temperature reaches 280 K (this should take about 15 to 20 minutes) reduce the pressure in the dewar to 0 bar using the manostat valve.
8. Set the heater power to zero voltage/percent.
9. Close the needle valve on the transfer tube.
10. Switch off the nozzle heater, (bottom switch located on back of the GFC1).
11. Close the outer shield valve.
12. Close the supply valve to room temperature helium gas.
13. Disconnect the 10mm tube from the transfer tube.
14. Close the storage dewar vent valve and open the pressure relief valve.
15. Remove the transfer tube from the storage dewar.

This procedure should stop any moisture getting inside the nozzle and heat exchanger. This should enable a trouble free start up.

6.7. Dealing with Malfunctions

Head going frosty	The vacuum has deteriorated. Pump out using the pump-out port (NW16 fitting) whilst the head is operating to see if the frost disappears. If the frost disappears, warm up and leak check the system. If no leak is found then pump out the system overnight. If a leak is found contact Oxford Diffraction for further advice.
Outer shield float goes to zero	Check helium pressure in the helium gas cylinder. If the pressure is too low warm up the system and replace the gas cylinder.

6.8. Emergency Procedures

Use the emergency procedure below:

- If there is a fire or any other emergency requiring the evacuation of personnel from the area

AND

- The procedure can be performed without endangering any persons' safety.

6.8.1. Stopping all Cryogen Flow to the Head

This procedure will stop all cryogen flow to the head immediately.

1. Vent the helium storage dewar through the Manostat valve.
2. Turn off the GFC1 pump.
3. Close the gas cylinder supply valve.

7. Maintenance

7.1. Introduction

> **WARNING**
> **Failure to perform scheduled maintenance tasks properly and at the correct intervals can affect the safety and performance of this system.**

> **WARNING**
> **Before performing any maintenance task, ensure that you have read and understood the HEALTH AND SAFETY INFORMATION at the beginning of this manual.**

Maintenance must be performed to ensure that the Helijet system continues to operate safely and reliably. This is detailed in the maintenance plan given below.

Maintenance that can be performed by the operator is limited to the routine maintenance tasks listed below.

7.2. Maintenance Plan

7.2.1. Before Using the Helijet

The vacuum space in both the LLT and the Helijet head initially require regular pumping depending upon usage. The vacuum space in the LLT has been baked during assembly and therefore should only require re-pumping every 6-12 months depending upon use.

To get the best possible results, pump the vacuum space in the Helijet head before use.

After about 6 months the intervals between pumping out should become longer as the outgassing on the internal components decreases.

7.2.2. Six Month Schedule

Pump out overnight the LLT through the port on the top of the helium dewar using a 70 l/s turbomolecular pump.

7.2.3. 10000 Hour Schedule

Under normal operating conditions the pump is maintenance free. The normal operating lifetime of the valves and diaphragm is >10000 operating hours. The valves and diaphragm are wearing parts and if the ultimate vacuum pressure is unobtainable the pump interior, valves and diaphragm must be cleaned and inspected for damage and cracks.

7.2.4. Five Year Schedule

Store the internal memory and change the battery after 5 years. The ITC has a battery back up RAM and the lifetime of the power source is generally 5 – 7 years.

8. Trouble Shooting

8.1. Fault Diagnosis

Symptom	Possible causes
Unable to obtain base temperature	1. Vacuum issue (air leak, internal leak poor initial vacuum) 2. Low flow from dewar 3. Partial blockage 4. Out of liquid helium in storage dewar 5. Nozzle shields not concentric
Temperature instability	1. Vacuum issue 2. Wrong PID values 3. Wrong ITC settings 4. Heater failure in heat exchanger
Incorrect temperature reading	1. Sensor calibration incorrect 2. Thermocouple failed
Condensation or ice on the outer nozzle	1. Heater in the nozzle not operating
Failure to control at any given set temperature above base temperature	1. Heater failure

8.2. Trouble Shooting Procedures

8.2.1. Blockages in the Transfer Tube

If you have persistent problems with blockages in the transfer tube you may be able to reduce the risk by altering the operating procedure as follows.

1. Before you start to lower the transfer tube leg into the storage dewar, connect the “From LLT” connection of the GFC1 to the side arm end of the transfer tube using a short length of rubber tube.

2. Use the diaphragm pump in the GFC1 to draw helium gas through the transfer tube until it is cold. Then, wearing thick gloves, quickly remove the rubber tube and insert the transfer tube into the side arm as described in the operating procedure.

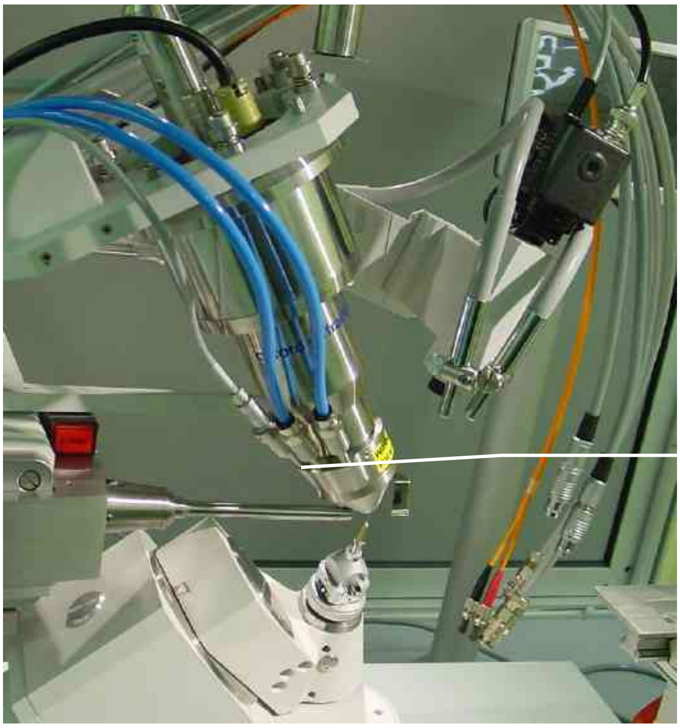
3. Connect the GFC1 “From LLT” tube to the normal exhaust port on the LLT and continue as described in the operating procedure.

If the head does not cool down there could be a problem with the PTFE seal. This is usually indicated by the helium flow and pressure NOT reducing on the inner shield flow when the knurled nut is first tightened. The flow rate will then drop again because of the impedance of the small tube in the cryostat (which has not yet cooled down). As this tube cools, the flow increases again, and after about 10 minutes it typically increases to 1.5 litres per hour. First try to reset the coupling as described in the transfer tube assembly. If this does not solve the problem there may be a blockage in the head or there could be ice formed on the face of the PTFE seal. If this is the case warm the transfer tube and Helijet head to room temperature and blow dry helium gas through the helium circuits until they are dry and free from any obstructions.

8.2.2. Changing the Heater in the Nozzle

Equipment required: 1 mm Allen key (Hexagonal wrench)
Soldering iron
Soft solder
Heat shrink (approximately 2 cm)
New heater

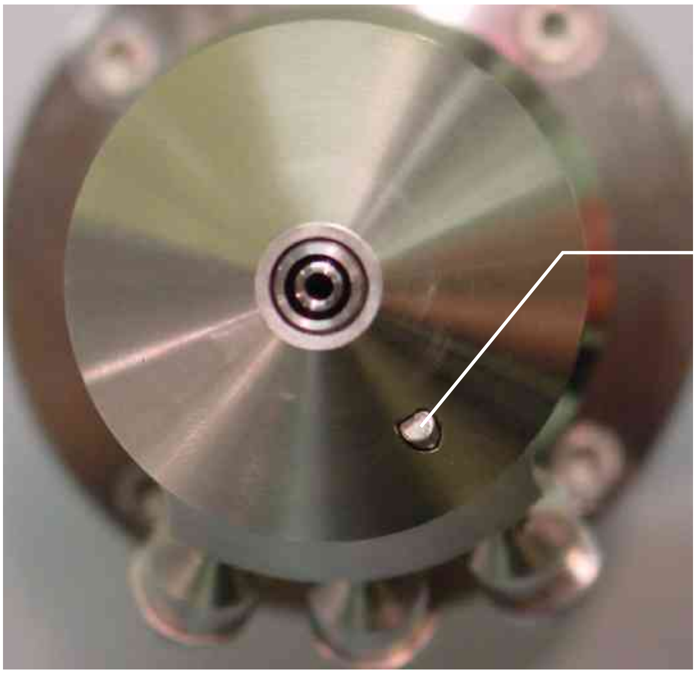
1. Undo the grub screw shown in the diagram below.



Grub screw

2. Slide out the 2-pin Fischer connector.

3. Unsolder the old heater and remove it. If it is stuck, push through the hole (shown below) in the nozzle from underneath.



4. Fit a new heater.

8.2.3. Re-aligning Nozzle Shields

Equipment required: 1.5 mm Allen key (Hexagonal wrench)

1. Loosen the three grub (set) screws around the outside of the nozzle. Do not undo the grub screws completely.

2. Adjust the position of the nozzle assembly until it is visually concentric.

3. Re-tighten the grub screws, gradually, whilst visually re-checking the alignment.

8.2.4. Checking and Changing the Heater

1. Check that the heater needs changing by measuring the resistance across pins 1 + 2 on the connector. The resistance should be about 80 ohms.

2. Contact Oxford Diffraction service department for advice about changing the heater.

8.2.5. Changing the Thermocouple

Contact Oxford Diffraction service department for advice about changing the thermocouple.

9. Spares

9.1. 0-rings

Size in mm		Size in inches		Item no. on the assembly drawing HA-00-00-000-C	Quantity per set	ODL Part No.
internal diameter	section diameter	internal diameter	section diameter			
61.6	2.62	2.425	0.103	37	1	MO-02-62-062-P
35	1.5	1.378	0.059	39 & 40	1	MO-01-50-035-P
29.97	2.62	1.180	0.103	38	1	MO-02-62-030-P
18.77	1.78	0.739	0.070	41	1	MO-01-78-019-P
12.7	2.62	0.500	0.103		1	MO-02-62-012-P
14	1.78	0.551	0.070	43	1	MO-01-78-014-P

Notes:
* three options are given for the same function
** there are also three O-rings in the vacuum valve in the He entry arm

9.2. Pump

Description	Part Number
Set of wearing parts	PK050 119-T

9.3. Fuses

Designators	Value	Package	Location
EA-10-00-004-A	230V AC - 0.8AT/250V	5x20mm	ITC502
EA-10-00-005-A	110V AC - 1.6AT/250V	5x20mm	ITC502
EA-10-00-018-A	230V AC - 1AT/250V	5x20mm	GFC1
EA-10-00-007-A	110V AC - 2AT/250V	5x20mm	GFC1
EA-10-00-004-A	250V AC – 0.8AT/250V	5x20mm	GFC1

9.4. Helijet Head

Description	Part Number
Nozzle heater (40V 20W)	EA-03-00-046-A
Heat exchanger and heater (40V 20W)	EA-03-00-047-A

9.5. Bulbs

Description	Part Number
GFC1 bulbs (60 V 20mA)	EA-04-00-017-A

10. Disposal Information

10.1. Helijet

Contact a waste disposal agency to ensure that the Helijet system and electronics are disposed of in accordance with any local and national regulations.

10.2. Third Party Equipment

Refer to third party manuals for information about disposing of third party equipment.

11. Additional Information

11.1. Third Party Information

Description	Manufacturer	Model Number	Type of Manual
Temperature controller	Oxford Instruments	ITC 502	Operator’s handbook
Liquid helium transfer tube	Oxford Instruments	LLT series	Operator’s handbook
ObjectBench	Oxford Instruments	Version 2.8.7	Operator’s handbook
Pump	Pfeiffer	MVP0202-3 AC	
Auto needle valve for ITC502 and ITC 503	Oxford Instruments		

11.2. Drawings

Drawing Number	Drawing Title
HA-00-00-007-C	Mounting plate
HA-00-00-008-C	Mounting plate
HA-09-00-001-C	Helijet head dimensions
EH-00-00-001-B	Helijet electrical diagram
EH-00-00-010-C	GFC1 – electrical diagram
EH-00-01-001-A	Helijet compensator cable
EH-00-01-002-A	Helijet thermocouple interface
EH-01-07-001-A	GFC1 – Supply cable
EH-02-02-002-A	Nozzle heater cable
EH-04-05-001-A	Helijet RS232C cable

12. CE Conformity notice

DECLARATION OF CONFORMITY

This Declaration of Conformity is suitable to the European Standard EN 45014, "General criteria for supplier' s declaration of conformity." The basis for the criteria has been found in international documentation, particularly in: ISO/IEC Guide 22, 1982, "Information on manufacturer's declaration of conformity with standards or other technical specifications."

Oxford Diffraction's liability under this declaration is limited to that set forth in the current Oxford Diffraction Terms and Conditions of Sale.

Applied Council Directive(s):
89/336/EEC Electromagnetic Compatibility Directive (EMC)
and amending directives 91/263/EEC, 92/31/EEC, 93/68/EEC.
73/23/EEC Low Voltage Directive, and amending directive 93/68/EEC

We, The Manufacturer:

Oxford Diffraction
Nuffield Way
Abingdon OX14 1RL

declare under our sole responsibility that the following equipment:

Helijet with Oxford Instruments Intelligent Temperature Controller (ITC) & Oxford Diffraction Gas Flow Controller (GFC)

Serial Number From: HAA-003/01

to which this declaration relates are in conformity with the relevant provisions of the following standard(s) or other normative document(s) when installed in conformance with the installation instructions contained in the product documentation:

EN 55022:1998 Class B for radiated and mains conducted emissions.
EN 50082-2:1999 for the following: Electrostatic Discharge immunity, Electric Fast Transient immunity, RF Conducted immunity, Surge immunity and Voltage dips immunity.

Pertinent sections of:
EN 61010-1, Safety requirements for electrical equipment for measurement, control and laboratory use Part 1: General requirements.

Technical Information is maintained at:
Oxford Diffraction
Nuffield Way
Abingdon OX14 1RL
United Kingdom

Last two digits of year of CE Marking (low Voltage Directive): 02

We, the undersigned, hereby declare that the product(s) specified above conforms to the listed directive(s) and standard(s).

Signature:

Full Name: Paul Loeffen
Position: Managing Director
Date: 12/07/02

Appendix 1 Helijet Short Guide

The short guide (on the next page) should only be used by experienced operators of the Helijet system. Inexperienced operators of the Helijet system should use the full operating instructions in section 6 of this manual.

The short guide can be printed out and kept near to the system. Its purpose is to serve as a quick reminder of the steps to be taken when operating the Helijet system.

Helijet Short Guide

Start Up

1. Ensure that all tubes are connected to the correct ports except the 10 mm tube, this should be connected after the transfer tube has been inserted into the storage dewar.

2. Close all 3 control valves and put the 3-way valve into the operation position (located on front panel of the GFC1).

3. Ensure that all electrical plugs are connected

4. Switch on the ITC and check that the ITC is giving a room temperature reading.

5. Turn on the nozzle heater, (bottom switch located on back of the GFC1).

6. Turn on the diaphragm pump located inside the GFC1, (top switch located on back of the GFC1), the pressure should go down to about 0mbar with no flow indicated on the flow gauge.

7. The temperature reading on the ITC should rise above 300 K; this is an indication that the nozzle heater is working correctly.

8. Open the needle valve on the transfer tube 4 turns.

9. Insert the 12mm diameter leg of the transfer tube into the helium storage dewar slowly, ensuring that the pressure in the dewar does not exceed 0.5bar, open the storage dewar pressure relief vent if required.

10. Connect the 10mm tube to the return flow on the transfer tube.

11. Set the pressure on the helium gas supply to 1.5bar.

12. Open the blue manostat valve and set the pressure in the helium storage dewar to 0.3bar. Ensure that the vent valve on storage dewar is closed.

13. Turn the 3-way valve to purge and set the flow on the outer shield flow gauge to 1.5 l/hr. Leave for 2 – 3 minutes. This will purge the GFC1 internal pipe work, pumping system and the inner nozzle.

14. Open the vent valve on the storage dewar.

15. Remove the protective cover from the end of the transfer tube and check that the white Teflon seal is clean, dry and in the correct location.

16. Check that there is a gas flow from the end of the transfer tube.

17. Insert the transfer tube into the sidearm of the Helijet head and engage locknut 1 turn.

18. Tighten the lock nut hand tight, this allows the helium to follow the correct path through the head.

19. Turn the 3-way valve to operation and set the flow on the outer shield flow gauge to 0.7l/hr and leave for 2 – 3 minutes. This will purge the outer nozzle.

20. After about 8 - 10 minutes the temperature should start to drop.

21. When the temperature reaches about 100 K, open the inner flow control needle valve slowly and set the pressure to 200mbar. This should set the inner shield flow to about 0.4l/hr.

22. The temperature should reach 15 K within 10 – 12 minutes from start.

23. When the base temperature of about 10 K is achieved reduce the pressure in the storage dewar to 0.2bar using the blue manostat valve. Re-adjust the outer shield flow to 0.7 l/hr.

24. The head should now be operational.

Operating System

1. With an inner shield flow setting of 200mbar the operating temperature range is between 15 – 75 K.

2. To control within this range use a PID setting of 2.8, 0.7, 0.0, although if the temperature becomes unstable increasing the P value will dampen any temperature oscillation

3. The PID settings can be adjusted by pressing the relevant button and simultaneously pressing the raise/lower button.

4. To set the temperature, press and hold the set button then adjust the set point by using the raise/lower buttons.

5. To obtain the set temperature, press the auto button on the heater section of the ITC front panel.

6. The temperature should become stable within about 5 minutes to +/- 0.2K however the time will depend upon the temperature jump.

7. If you require to work above 75 K it is possible to increase the upper limit to about 100 K by reducing the pressure on the helium storage dewar to 0.125 bar as indicated on the outer shield pressure gauge and reducing the pressure on the inner shield vacuum gauge to 100 mbar.

8. You should not go lower than 100mbar on the inner shield vacuum gauge because that will cause the cold stream to stall and it will then be possible for moisture to form inside the head.

9. It is possible to increase the temperature a little further by closing the needle valve located on the transfer tube 3 ½ turns.

Warm-Up

1. Set the temperature to 280 K and ensure that the ITC is in auto mode (this will turn the heater to a maximum output of 40.0V).

2. Close the inner shield valve.

3. Close the needle valve located on the transfer tube 3 ½ turns so that it remains ½ a turn open.

4. Reduce the dewar pressure to 0.05 bar.

5. Turn off the pump in the GFC1.

6. Vent the pump to an atmosphere of helium by using the operation/purge value in the purge position.

7. When the temperature reaches 280 K (should take about 15 minutes) reduce the pressure in the dewar to 0 bar using the manostat valve.

8. Set the heater power to zero voltage/percent.

9. Close the needle valve on the transfer tube.

10. Switch off the nozzle heater, (bottom switch located on back of the GFC1).

11. Close the outer shield valve.

12. Close the supply valve to room temperature helium gas.

13. Disconnect the 10mm tube from the transfer tube.

14. Close the storage dewar vent valve and open the pressure relief valve.

15. Remove the transfer tube from the storage dewar.